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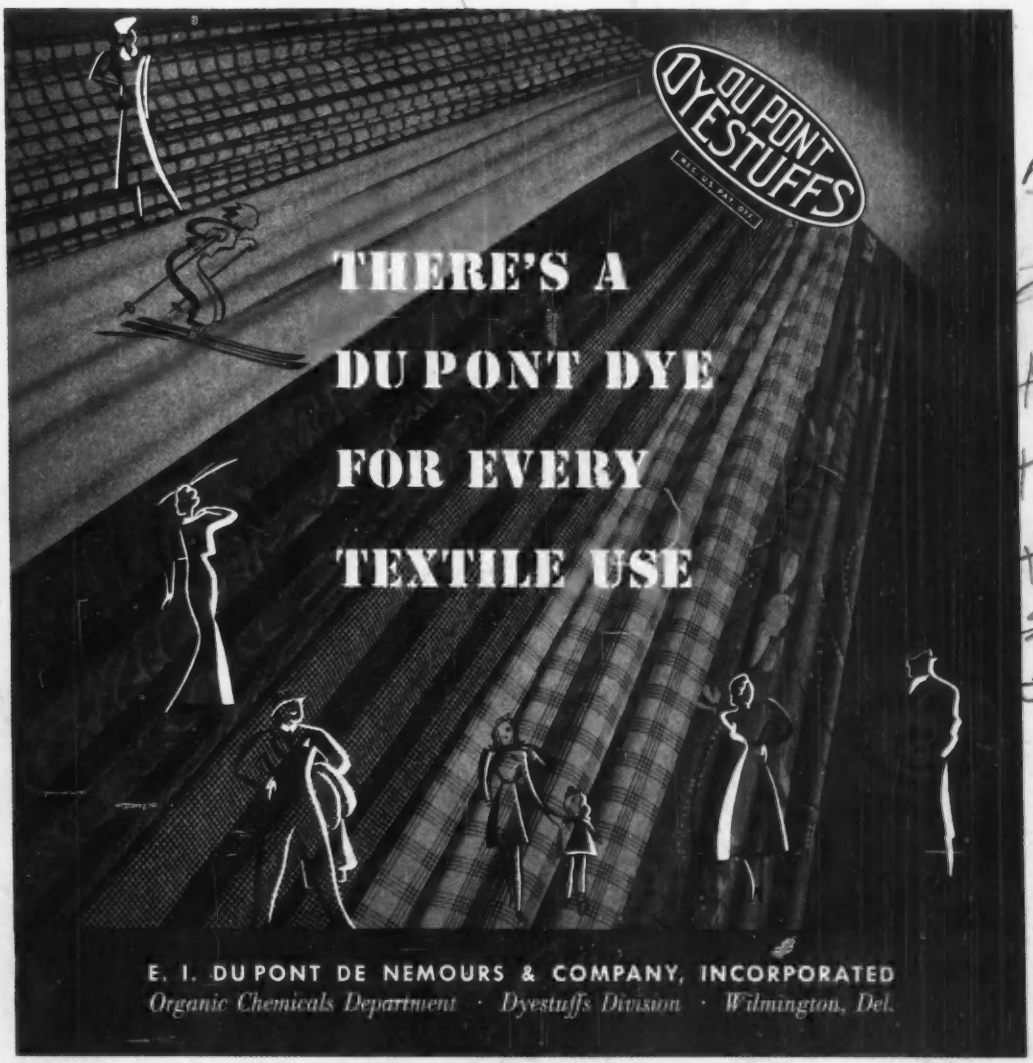
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AMERICAN DYESTUFF REPORTER

Official Journal

The Adsorption of Alcohols by Wool*

A: Studies on the System: Ethyl Alcohol-Wool

FRANK J. RIZZO and HUGH CHRISTISON

1. GENERAL

RECENTLY, occasion arose to study the properties of a certain hydrocarbon solvent upon wool substance, special attention being devoted to the question of its adsorption. For the sake of comparison as well as of control, reference was made to the action of water and of alcohol on wool the latter of which Mease[1] found to be adsorbed to the extent of 1.8 per cent, even at temperatures well above the boiling point of the alcohol, although readily and quantitatively removed by water.

In the problem at hand, it was deemed advisable to avoid all heating operations. With due attention to the phenomenon of hysteresis in wool, the samples were brought to a conditioned weight by alternating successively between a relatively dry atmosphere and a relatively moist one, as compared with the room at the moment, finishing off in air of normal relative humidity, namely, the laboratory atmosphere. The whole operation consumed about six hours. The samples were then dipped in the solvent and reconditioned as before, all under proper control. Much to the writers' surprise, all the samples emerged from these operations unaltered in weight. Repetition showed the same results.

These results were most disturbing in view of Mease work. The logical reasoning was, either (a) the alcohol does not replace bound moisture or (b) the moisture of the atmosphere replaces the alcohol. The second of these assumptions appeared the more probable, particularly when one considers the manner in which the samples were conditioned. It received support also from Mease' findings on the ease of removal of the alcohol by water. If such be the case, it was reasonable to expect that, after any period of exposure to an atmosphere of known characteristics of a sample of wool bearing adsorbed alcohol, the percentage of alcohol still remaining on the wool might be proportionate to the elapsed time during exposure and the relative humidity of the surrounding medium.

Conceding that Mease had very specifically outlined the conditions of operation under which the adsorption took place, but at the same time realizing the probability of alteration of known procedural schemes in busy mill laboratories, it was thought desirable that this problem be better understood.

2. METHODS

The samples of wool which were used in this work consisted of skeins of loosely spun yarn made from 64's grade Territory wool. The preparatory operations consisted of scouring in the conventional soap and soda solution, acidification with dilute acetic acid, rinsing in lukewarm water to neutrality, drying, extraction with ether and alcohol and finally rinsing in warm distilled water. All the skeins were then conditioned in the air as above and weighed immediately in tared, covered tins. These skeins served as working material for our four sets of experiments:

(I) The air-conditioned yarn was immersed in the solvent directly, excess solvent squeezed out, and then dried in the conditioning oven at 105-110° C.

(II) The air-conditioned yarn was immersed in the solvent directly, excess solvent squeezed out, reconditioned in the air as above, and oven-dried at 105-110° C.

(III) The air-conditioned skein was oven-dried at 105-110° C., taken directly from the oven and plunged immediately into the solvent, excess solvent squeezed out, and redried in the oven as before.

(IV) The air-conditioned skein was oven-dried at 105-110° C., taken directly from the oven and plunged immediately into the solvent, excess solvent squeezed out, air-conditioned again and redried in the oven at 105-110° C.

Drying time was in all cases about two hours. This is the length of time usually found necessary for a sample of wool containing a normal amount of regain to come to equilibrium with the conditions within the drying chamber. It was realized that the solvent might be removed sooner than moisture, but the same length of time was allowed for this, provided equilibrium had been attained. Where constancy was not realized in this set time, the period was prolonged until such was reached.

Variations in the above procedure were made only to the extent that in some of the experiments water was used as solvent; in some, the samples coming out of the oven after drying in-the-solvent were immersed in water, rinsed thoroughly and reinserted in the oven; in other cases, samples coming from the oven after drying in-the-solvent were air-conditioned as above before reinsertion in the oven.

3. RESULTS

The first experiment was designed to check the work of Mease using the method he outlined in his paper (procedure III above). Using absolute alcohol and a long

*A Contribution from the Laboratory of the Arlington Mills, Lawrence, Mass.

[1]Ind. & Eng. Chem. Anal. Ed. 1933, 5, 317.

list of non-hydroxylic solvents, a duplication of Mease' results was obtained. The alcohol was adsorbed to the extent of 1.8 per cent under drying conditions of 105-110° C. and was quantitatively removed by water. Results for the other solvents are not shown, but suffice to say that the work not only substantiates Mease' findings but is also in line with the observations of Speakman[2] during a study of load-extension properties of single dried fibers immersed in organic solvents. It seems safe, accordingly, to make the assertion that all solvents, other than actual alcoholic hydroxylic compounds and those potentially so either by hydrate formation, enolization and the like, will be inappreciably adsorbed.

Having established the proper conditions for the determination of the degree of adsorption as measured for ethyl alcohol, we proceeded to test our surmises by the series of tests outlined above. Because of the greater accessibility of No. 30 Formula Alcohol*, which is the usual azeotropic mixture of water and ethyl alcohol denatured with methyl alcohol, we proposed to conduct the experiments with this. Before doing so, assurance was necessary that the few percent of moisture in this mixture would not materially affect the results. Accordingly, a mixture of absolute ethyl alcohol and water was made to approximate this. The mixture finally used was 4 per cent by volume of water and 96 per cent absolute ethyl alcohol. The results with this solvent were so close to those with the absolute ethyl alcohol alone when applied to oven-dried samples that no objection could be envisioned to the use of the No. 30 Alcohol. The only factor still remaining unconsidered was the presence of the small percentage of methyl alcohol. It was the findings with this solvent which subsequently actuated the work presented in part B of this paper. The outcome of the four experiments outlined before were:

Table I

Method I	1.9%	Adsorption
Method II	0	Adsorption
Method III	2.2	Adsorption
Method IV	0	Adsorption

A glance at the table reveals that in the case where the alcohol was applied to oven-dried samples not subsequently exposed to an atmosphere of high relative humidity (Exp. III), the amount of bound or adsorbed alcohol is very definite, in the case of the No. 30 Alcohol having been found to be 0.4 per cent higher than with absolute ethyl alcohol. It is further observable that, if the wool already contains a normal amount of moisture when the alcohol is applied, the alcohol will replace the moisture from the wool and will itself be adsorbed at drying temperatures in a very definite amount, although smaller in value than with oven dry wool. In other words, combining the information which Methods I and III present us, it can be said that, starting with samples of varying degree of moisture from 0 per cent up, the effect of the moisture at the low concentrations (regains of say up to 5 per cent) will be relatively negligible, but beyond this point the effect of the regain will assume greater and greater significance. The results obtained with Methods II and IV further reveal that any exposure of the wool to the atmosphere after the alcohol treatment tends to reduce the amount of the adsorbed alcohol and if left long enough or if the relative humidity of the atmosphere in which the samples are suspended is great enough, the loss of the alcohol is complete. To express this in more scientific language, the percentage of bound or adsorbed alcohol to be found on wool after exposure to an atmosphere will be a function of the elapsed time during exposure, the atmospheric relative humidity and the temperature of the atmospheric environment.

It should be noted that the combination of initial regain in the wool and exposure of the wool to the atmosphere after treatment with the alcohol increases considerably the rate at which the alcohol is lost from the wool. All the experimental evidence obtained points conclusively to the assertions above expounded. There is no reason to believe that, although the results with the No. 30 Alcohol are somewhat higher than with the absolute ethyl alcohol, as regards its reactions to moisture conditions of the surrounding medium, etc., the use of the absolute ethyl alcohol would have called for any different conclusions.

The anomalous behavior of alcohol being held so tenaciously by the wool substance yet so readily removed by moisture, which is itself seemingly so readily removed at temperatures approximating its boiling point, may be noted. Speakman[2] had already commented on this phase of the work, although under dissimilar conditions, stating that the action of the hydroxy compounds was to open the coordinate link with the subsequent formation of esters with the liberated carboxyl groups. Parenthetically, it may be noted that the wool should show a higher isoelectric point, although its determination may be difficult. Conceding the correctness of this interpretation, the action of the water is, therefore, hydrolytic, permitting the re-establishment of the coordinate link. However, operating at elevated temperatures plus the presence of considerable aldehyde fumes in the drying chamber following the admission of the alcohol-soaked samples raised questions which must of necessity be answered.

In the case of the aldehyde, this might turn up either in some polymeric form impregnating the fiber substance, but still water soluble, or as a substituted formol-amine reaction product, this reaction between aldehyde and amine being well known. To distinguish between ester formation, substituted aldehyde-amine and condensation product a few experiments were conducted.

If the condensation material were produced, it should be recoverable in water and identified after depolymerization by oximation or any other of the typical aldehyde

*A U. S. Government Formula most readily accessible to the American laboratory worker.

[2]Trans. Faraday Soc. 25, 169-76, 1929; ibid, 26, 61-9, 1930.

identification procedures. With the aldehyde-amine, the isoelectric point of the wool should change making it more acid. No water soluble condensation product was obtainable; no noticeable change in the isoelectric point to the acid region was to be noted. Despite this conclusive evidence, it was decided to attack the problem from still another angle. Accordingly, wool was dried in vacuum, the flask containing the wool being immersed in a boiling water bath. Aldehyde-free alcohol was then drawn into the flask, the wool allowed to become saturated, and the alcohol then withdrawn under the same operating conditions as in the initial drying. This method proved beyond all doubt that the alcohol was adsorbed as such and hence there is no reason to question ester formation as expounded by Speakman. It is interesting to note that this procedure gave within the limits of error a duplication of the results obtained in the oven for both the absolute alcohol and for the No. 30 Alcohol.

B: The System: Alcohols—Wool

The unpredictable behavior of the No. 30 Formula Alcohol in the experiments outlined in the first part of this paper were of such theoretical, if not fully as practical value that it was decided to look further into the problem. There were two other problems to clarify at the same time. First, conditions of experimentation sometimes require the use of an alcohol other than ethyl alcohol. A few of the more useful ones were, therefore, tested for their degree of adsorption. Secondly, although Speakman[2, 3] demonstrated that alcohols having a molecular size greater than that of n-propyl alcohol cannot enter the fiber capillaries of clean dry fibers, his work was done at room temperatures and in atmospheres of the respective alcohol vapors. It was consequently of interest to see what would happen at the higher temperatures, especially in the region of the normal drying range, 105-110° C.

1. Mixtures of Methyl and Ethyl

Two thoughts presented themselves with regard to the behavior of the No. 30 Formula Alcohol. The methyl alcohol may be adsorbed independently of the ethyl alcohol, the latter being somewhat preferential, or, otherwise, the presence of the smaller molecular type aids the introduction of the larger.

Speakman[3] in a study of the micelle structure of the wool keratin by the use of load-extension data on dried fibers when immersed in alcohols had already come to the conclusion that any alcohol of greater molecular size than n-propyl could be made to enter the capillary structure by taking advantage of the previously observed swelling phenomenon in the presence of the smaller molecules. By the use of methyl alcohol-octyl alcohol mixtures, he was able to demonstrate the introduction into the fiber capillaries of the large octyl alcohol molecule, which by itself has not this power. In our own work, it has been possible to demonstrate that isoamyl alcohol, which is unadsorbed at drying temperature, when mixed in equal volume with methyl alcohol, shows an adsorption of better than 8 per cent. It appeared, therefore, that the methyl-ethyl mixture in the No. 30 Alcohol was functioning in much the same manner, probably indicating that the methyl was assisting the ethyl into capillary spaces not available to it under normal conditions. If this be so, we should expect this sort of behavior to reach a maximum with a mixture of the two alcohols low in the methyl alcohol concentration. As the methyl alcohol concentration increased we should expect to more and more approach the condition of pure methyl, never quite attaining it so long as ethyl alcohol were present. The results for several mixtures of these two follow:

Table II

% Methyl in Methyl-Ethyl Mixture	*% Total Absorption*
1%	1.8%
10%	2.2%
20%	1.8%
30%	1.7%
50%	1.5%
80%	1.3%
100%	1.2%

Thus early in the series does the role of the smaller molecule as pilot make itself manifest.

2. Adsorption of Aliphatic Alcohols

The results reported herewith were all obtained using Method III above. In the case of some of the higher alcohols, whose vapors are noxious and disagreeable, the greater part of the vapors were removed in vacuum at 100° C. The alcohols used were tested for traces of aldehyde with hydroxylamine. Utilizing the findings of Speakman on the adsorption of higher molecular alcohols in the presence of the smaller, as well as the results here reported for methyl-ethyl mixtures, traces of the lower alcohols were removed as part of the purification scheme by soaking oven-dried wool in the alcohol and then recovering the alcohol in vacuum. By this means, traces of the low molecular weight alcohols were retained in the dried wool.

Table III

Methyl Alcohol	1.2%	Adsorbed
Ethyl Alcohol	1.8%	Adsorbed
Isopropyl Alcohol	0	Adsorbed
n-Butyl Alcohol	0	Adsorbed
Isobutyl Alcohol	0	Adsorbed
tert.-Butyl Alcohol	0	Adsorbed
Isoamyl Alcohol	0	Adsorbed
50% Methyl Alcohol) + 50% Isoamyl Alcohol)	8.1%	Adsorbed

Proc. Roy. Soc. A132, 167-191, 1931.
[2]Nature 126, No. 3180, 565, 1930.

It is interesting to note that, if we compare the relative adsorption of ethyl and methyl alcohols with their respective molecular weights, we find in both instances a definite ratio. Assuming these same conditions to hold in the case of water, considered as the monohydrate, a value of 0.7 per cent is found for the adsorption of water under the conditions of the average conditioning oven. This seemingly represents the amount of moisture which remains in wool under normal drying conditions and may well be the one factor which stands between stability and decomposition. It shows that if we heat the wool much above 110° C. to remove this moisture, as we do so we yellow the fiber, producing breakdown.

3. Developed Hydroxy Compounds

Under this category it was proposed to study a very common laboratory solvent, acetone. While this represents the oxidation product of isopropyl alcohol, our interest in this was not excited, due to the fact that the isopropyl alcohol was so poorly adsorbed. However, Acetone, like most carbonyl compounds having active hydrogen in the alpha position to the carbonyl group, can show enolization, that is, under certain conditions it acts as though it were an alcohol:

$$CH_3—\underset{\underset{O}{\|}}{C}—CH_3 \quad \rightleftharpoons \quad CH_3—\underset{\underset{OH}{|}}{C}—CH_2$$

No adsorption was expected in the case of the solvent on dry wool because conditions would be thus ideal for its behavior as a non-polar compound, and furthermore the results with isopropyl alcohol showed that the molecular size of the acetone molecule would prevent its passage into the fiber capillaries. Using alcohol-free acetone on dried wool and distilling off the solvent in vacuum, no effect was observed, that is, acetone on dry wool in the absence of air shows no adsorption as such. If the dried wool is soaked in acetone and then oven-dried in the usual way, there is a large gain in t. The wool becomes permanently yellow, and the isoelectric point changes to the lower end of the scale, indicating that a reaction has taken place between the amino groups of the wool and the acetone, thus:

$$\diagdown\!\!\diagup C—NH_2 + O{:}C(CH_3)_2 \longrightarrow \diagdown\!\!\diagup C—N{:}C(CH_3)_2$$

This merely represents a realization of the reaction of a carbonyl group with the amino group, which was postulated for the aldehyde and the amino group in an earlier part of this paper. Now, if the acetone be applied to moist wool, or if the acetone contains a small amount of moisture when applied to dried wool, and the drying after the solvent is conducted in the absence of air, as in vacuum, a small but noticeable degree of adsorption can be demonstrated. The observations made were in the neighborhood of 0.5 per cent. The mechanism outlined above must therefore take place in order to show this amount. It is also obvious from the reaction of the carbonyl group with the amino group obtained above, that the influence of the oxygen in the atmosphere may have quite a marked effect on the behavior of the solvent at the keratin-air interfaces. The presence of the moisture served a twofold purpose in preparing the fiber capillaries for the entrance of the enolic form as well as facilitating the enolization.

DISCUSSION

The most noticeable feature perhaps is that in so far as the size of the capillaries (and this would apply also to those cases where entrance was made possible by the swelling action of the easily adsorbed solvents) will permit the molecule admittance to the inner structure, the degree of adsorption rises somewhat proportionately to the molecular weight and thus incidently to the boiling point. Now all the work herein reported indicates that the adsorption compound between alcohols and wool is an unstable entity, the whole tending to revert to a condition where the coordinate link is unbroken. The adsorption compound is presumably an ester, the hydrolysis of which will depend to a very large extent on the solubility of the alcohol in water. Where the adsorbed alcohol is large in comparison with the normal size of the capillaries and where the solubility in water is negligible a problem presents itself. Now it has been stated that the normal and stable condition of the wool is that at which all the coordinate linkages are intact. So long as any alcohol is present this condition does not exist, but as the molecular size of the adsorbed alcohol gets smaller and smaller the wool approaches nearer and nearer to the ideal state, so that we find a replacement taking place with an alcohol of large molecular size being removed by one of smaller dimensions. Of course, we are dealing with esters, and the extent of this removal can reasonably be expected to depend upon the concentration of the smaller alcohol, the percentage of the larger alcohol in solution in the lower, and the temperature at which the reaction takes place. In other words, we have here just another equilibrium reaction.

All this has a very practical value. It has long been known, that the extract from wool with alcohol contains among other things of undetermined nature, a certain percentage of alcoholic material, presumably cholesterol and isocholesterol. It is significant that this material, while quite soluble in ether in the free state, is extremely insensitive to this reagent in the preliminary extraction which usually precedes the alcohol treatment. These alcohols are readily removed by the ethyl alcohol extraction. Apparently, the mechanism here is: The alcohol used in the extraction replaces the cholesterol in the ester, the excess alcohol rendering its removal from the fiber easy by its swelling action on the fiber capillaries and its solubilizing action on the cholesterol itself.

It will of course occur to anyone that the cholesterol molecule is quite large and a question may arise as to how this originally found its way into the fiber capillary spaces and thus into combination. Cholesterol is a constituent

of wool fat, which contains among other things a certain percentage of soaps and salts, along with appreciable quantities of Nitrogenous Compounds. These salts and the moisture in the wool will, of course, cause considerable swelling of the wool fiber, probably as far as possible without disrupting its structure. Under such conditions the adsorption of cholesterol and other high molecular alcohols would be ideal. There is also the possibility that capillary conditions in the young fiber are different from those in the full grown.

It will also occur to many, that in the conventional cleaning operation, soap and alkali are used, so that swelling of the fiber would occur as well as saponification of any ester between an adsorbed alcohol and the amino acid carboxyl groups. Why it should be that the cholesterol liberated is not removed, is not revealed by our work, except that it may be the removal of any material from the fiber capillaries is not mechanical but physico-chemical, that to be removed it must be solubilized. In the washoff process following scouring, the alkali tends to remove rapidly, thus making it possible for the cholesterol to recombine to form the ester.

ACKNOWLEDGMENT

We wish to take this opportunity to express our appreciation to Mr. C. L. Nutting of this laboratory for his comments and suggestions on the work.

SUMMARY

1. Ethyl alcohol is adsorbed by wool to the extent of 1.8 per cent at the drying temperature, if applied to oven-dried material.

2. If applied to wool of normal regain, the alcohol replaces adsorbed moisture and is itself adsorbed.

3. Wool carrying adsorbed alcohol and exposed to the atmosphere loses the alcohol, due to the effect of the moisture in hydrolyzing the ester formed by the adsorbed alcohol with the carboxyl groups liberated by breaking the coordinate linkage.

4. Mixtures of methyl and ethyl alcohols show an increased adsorption, over that with ethyl alcohol alone, when concentrations of methyl alcohol are low and then a decreasing adsorption as the methyl alcohol concentration rises.

5. It is found that alcohols greater in molecular size than isopropyl are not adsorbed at the temperature of the conditioning oven.

6. Methyl alcohol is adsorbed by wool to the extent of 1.2 per cent when applied to oven-dried material.

7. Non-hydroxylic compounds, in fact all non-polar compounds, and those incapable of reverting to such, are not adsorbed by wool.

8. The enol form of acetone has been shown to be adsorbed to a small extent when acetone is applied to wool carrying a normal regain.

9. This study has shown a definite relationship between the percentage adsorption of methyl and ethyl alcohols and their molecular weights. Assuming a similar relationship for monohydrol, water, calculation shows that wool retains 0.7 per cent moisture in the average conditioning oven. It is suggested that this insures stability of the wool under those conditions.

10. The interesting case of the presence of adsorbed cholesterol in scoured wool is discussed.

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[1]Mease, R. T., Ind. & Eng. Chem. Anal. Ed. 1933, 5, No.5, 317.
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What Can the Mill Man Expect from Fundamental Research?*

MILTON HARRIS†

THERE has been a tendency of the practical man in the mill to discuss the results of fundamental laboratory experiments with the statement, "this is all very interesting but I don't see where it fits into my picture". This statement arises from the fact that only the immediate practical applications of chemistry are considered, rather than the theoretical principles, and is understandable, especially since the achievements in fundamental research are generally not as apparent as are the developments in the industrial laboratory and cannot be directly translated in terms of lower costs or extended uses.

This talk is not designed primarily to answer the above statement but a few of the points to be discussed have considerable bearing upon it. Its main purpose is to present a few examples which demonstrate the interrelation of the fundamental and the practical sciences and in doing this, it is hoped to not only make clear what the mill man can expect from fundamental research, but also to point

*Presented at Annual Meeting, Philadelphia, Dec. 4th.

†Research associate at the National Bureau of Standards for the American Association of Textile Chemists and Colorists.

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out what the role of the investigator in textile research should be.

The textile industry has long been a major one and yet with the exception of the development of synthetic fibers, there have been few important changes in the industry in an era in which most industries, as a result of scientific developments, have gone ahead by leaps and bounds. And it must be said that the synthetic fiber industry received most of its early impetus from without rather than from within the active textile industry. It is a well known fact that when rayon was first put on the market, a committee appointed by silk manufacturers to study its possibilities, declared it a passing fad. The reason for this unfortunate conclusion was probably attributable in part to a lack of understanding of the possibilities of scientific research. But even today, similar decisions are constantly being made. I have seen textile people laugh at lanital, a synthetic substitute for wool, and yet I feel sure that it is probably a very much superior fiber today than was the rayon of not too many years ago. And even though I feel that the extremely complex chemical and physical properties of wool will make it the most difficult of the fibers to duplicate synthetically, it would be as much a folly to now disregard synthetic fibers which will simulate wool as it was for the silk manufacturers of a few years ago to disregard rayon. Synthetic fibers are products of scientific research and are no doubt destined to become more and more important. Two choices remain then for the manufacturers who utilize the naturally-occurring fibers; namely, to improve the natural fiber which can only come about by a better understanding of the more fundamental nature of the material; or to study the properties of the synthetic fiber so that it can be utilized to its greatest advantage, if and when it must partially or wholly supplant the naturally occurring material. Both of these ends can only be accomplished by research.

The textile industry does not have to look far for examples of what research has done for other industries. Probably no single factor was more instrumental in pulling us out of the depression of 1921 than was the automobile, a product of research. And after our most recent depression, the industries which have made the most recovery are with few exceptions, those industries built on research. It is especially interesting to note that ten of eleven of our largest chemical companies paid common dividends throughout the depression. And in nearly every case the answer has been research resulting in the development of new methods, improved techniques, and new products.

If for the moment it is assumed that research is important to the future of the textile industry, the question arises as to whether emphasis should be placed on fundamental research. As it has been previously mentioned, the most obvious developments arise from the practical applications but as is very often the case, the fundamental discoveries precede the practical applications. There are many examples today of whole industries which have been built on the results of researches which were started with no practical end in view. Irving Langmuir's studies on surface chemistry led to the development of greatly improved light bulbs, new methods of producing high vacuums, new developments in the field of radio engineering, and the development of the atomic hydrogen torch. The late Father Nieuwland's work at Notre Dame University on the condensation products of acetylene furnished the fundamental background for the development of the synthetic rubber, Neoprene, by the du Pont Company.

Unfortunately, there is no large, well-organized scientific group engaged in the study of textiles and textile materials. Some day, soon we hope, there will be such a group and it will be a fortunate one indeed. For it, there will be available a great mass of knowledge which is being accumulated in every branch of science, and some of which is waiting to be properly interpreted so that it may be put into circulation for the textile industry. The successful textile research man of the future will be the one who is acquainted with many fields of science. The boundaries of science are rapidly losing their significance and today there are countless examples of the results of one science being applied to another. The most recent postulate as to the structure of the proteins has come from a mathematician, while a physicist, as a result of studying sound waves, has contributed much to chemistry.

In our own work, on the chemistry of wool, we have found many parallelisms between the fundamental chemistry of this textile material and that of many biological substances which are now so important in studies relating to cancer, arthritis and diabetes research. For example, insulin, which is used in the treatment of diabetes, is a sulfur containing protein having many chemical properties similar to those of wool. We have found that oxidizing and reducing agents, alkalies, and exposure to strong light change the state of the sulfur in wool, which either causes direct degradation of the fiber or produces incipient damage which is greatly aggravated in the subsequent processing. Similarly, the same treatments change the state of the sulfur in insulin and destroy its physiological activity.

Perhaps the best example of the exchange of fundamental data of two such different fields as textiles and medicine resulted from our studies on the oxidation of wool. In the early work, we showed that careful oxidation of wool did not appear to damage it physically. However, its susceptibility to wet processing was greatly increased, and this fact puzzled us greatly. About the same time, two biochemists, Drs. Toennies and Lavine of the Research Institute of the Landenau Hospital of Philadelphia were engaged in the study of the oxidation of the amino acid cystine, as part of a program relating to cancer research. They found that oxygen would add directly to the disulfide group and that the stability of the oxidized derivatives decreased as the number of the oxygen atoms taken up increased. This gave us a much needed clue. Because of the drastic treatments which must be utilized

in order to break wool down to its component parts, it was impossible to isolate partially oxidized cystine derivatives from a wool hydrolysate, but it was possible to obtain evidence by no less than six different methods of the existence of such compounds in wool. With the knowledge that the sulfur in wool was readily oxidized and by knowing the properties of the partially oxidized compounds of cystine, it was a relatively simple matter to predict the properties of oxidized wool. The susceptibility to alkalies and alkaline reagents is greatly increased and as a result of this, the alkali-solubility test for determining the extent of oxidation of wool was developed. Some of the partially oxidized groups in wool are oxidizing agents and this fact has been utilized in developing another test for distinguishing between oxidized and untreated wool.

The above example is one of a great many which can be cited to illustrate the relation of textile chemistry to other fields of science. There is a wealth of scientific data now available and there is an ample supply of scientific investigators who are constantly furnishing new data in all fields of research. But there is lacking in the textile industry an organized group of scientists who understand the fundamental concepts of chemistry, who can assimilate the available material and evaluate it, and finally, who can translate it into the language of the mill man. The need of such a group is imperative since only by understanding this scientific knowledge can the mill man hope to utilize it.

—♦ ♦—

Methods of Determining Oil, Soap and Other

Extraneous Matter in Textile Materials*

Project 9-37

II — Investigation of the Nature of the Material Obtained by the Extraction of Wool with Ethyl Alcohol

BERTIL A. RYBERG†

INTRODUCTION

THE report covering Part I of this project, published in the Special Research Number of the AMERICAN DYESTUFF REPORTER, July 26, 1937, covered a partial analysis of this alcohol extract only, and left a number of unexplained points to clear up. This first work was done on material obtained by a large number of extractions on a small scale. It was thought that the continued boiling of this material in alcohol might result in a material of different composition from that obtained in a single extraction.

A large scale Soxhlet extractor was designed which would hold six pounds of wool top. In such an extractor it would be possible to obtain enough alcohol extractable material in one complete extraction, to make an analysis of it and thus determine its composition. The composition so determined would conform to that obtained in the conventional 250 ml. Soxhlet.

Figure 1 is a photograph of this large scale extractor, which is made of monel metal and is separable into three parts. It has one hundred times the capacity of the 250 ml. size Soxhlet and stands eight feet in height. Figure 2 shows a cross-sectional diagram of it, indicating the various parts and scheme of operation. It differs from the conventional Soxhlet in that the vapor tube passes up through the center of the extractor and the siphon is also contained in it. It is fitted with a gauge glass in order that the progress of the extraction may be followed.

The condenser was designed as such in order that a large amount of condensing surface be available for use with extremely low boiling solvents such as ethyl ether. The siphon tube was extended to within two inches of the bottom of the reservoir in order that it be continuously sealed and thus assure proper siphoning in a minimum of time.

Materials

Two lots of fine Australian wool in top form, were obtained, having been prepared as follows:—

Lot 1. Wool cleaned from grease etc. by the Solvent method, rinsed in warm water, carded and dry combed. This lot was prepared without using any oil, soap or alkali during processing, other than those naturally present in virgin wool.

*This project is supported by a grant from the Textile Foundation to the American Association of Textile Chemists and Colorists. This report was presented in abstract form at the Annual Meeting, Philadelphia, Pa., December 4, 1937.

†Research Associate of the American Association of Textile Chemists & Colorists, working at the Lowell Textile Institute, under the direction of the Sub-Committee on Methods of Determining Oil, Soap and Other Extraneous Matter in Textile Materials, Hugh Christison, Chairman, Leslie Christison, John Dalton, Alex. Morrison, Roland E. Derby, Harold W. Leitch, Clarence L. Nutting, Louis A. Olney, John H. Skinkle.

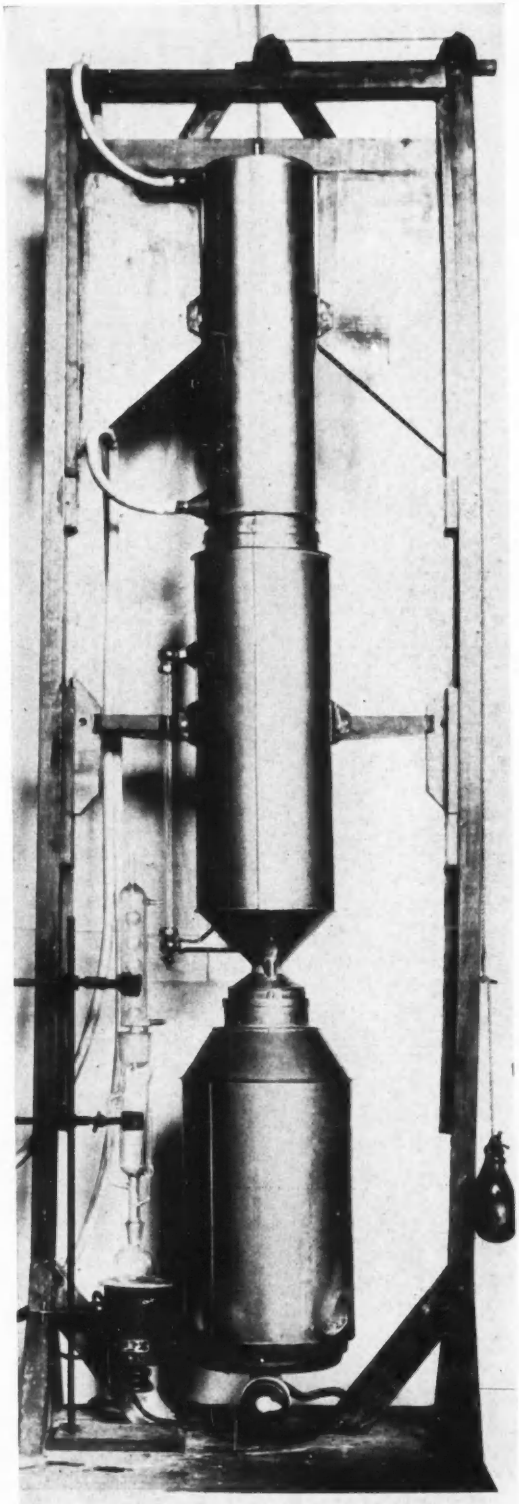


Fig. 1

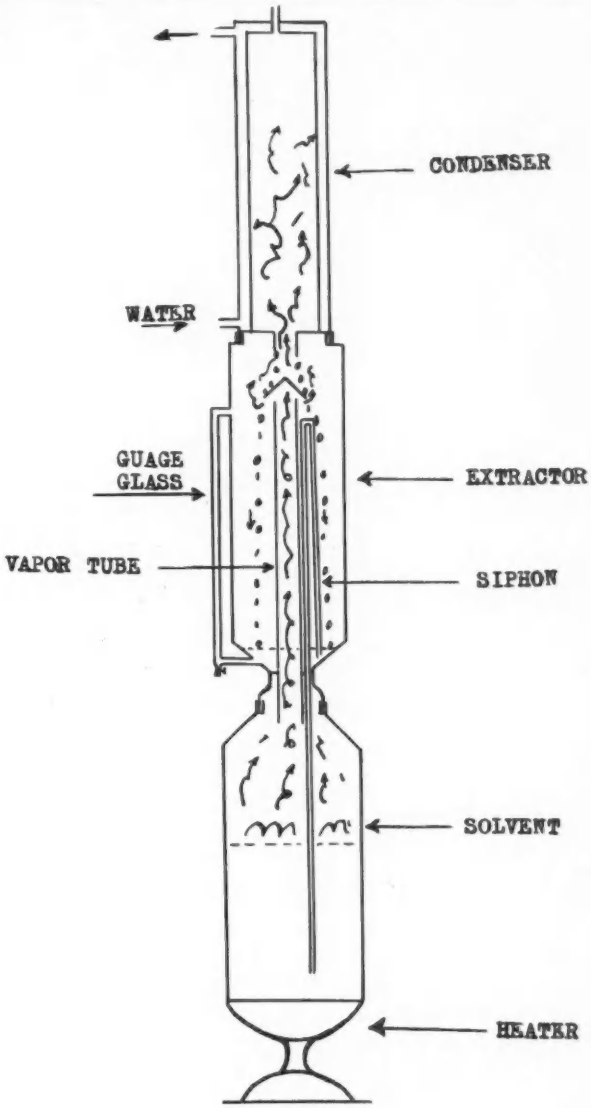


Fig. 2

Lot 2. This lot was prepared by the conventional method of scouring, using a Red Oil Soda Soap and sodium carbonate. It was carded and combed with the addition of oil, then backwashed with a sulfated alcohol.

These two lots of wool therefore, are representative of the two general methods used for cleaning wool.

Ethyl Ether, Anhydrous Analytical Reagent complying with the A.C.S. Specifications and containing not over 0.02 per cent alcohol.

Ethyl Alcohol, ninety-five per cent.

Methods

The methods used in this work are in most cases modifications of existing methods, these modifications having been made in order to adapt them to the nature of the material being investigated.

The wool was prepared for the alcohol extraction by extracting twelve times with anhydrous ethyl ether and thoroughly air drying. The wool was then replaced in the extractor and extracted twelve times with 95 per cent ethyl alcohol. The time cycle for a complete filling and emptying of the extractor was found to be approximately three times that required by the conventional 250 ml. glass Soxhlet.

The alcohol extract obtained was reduced to a definite volume, say one liter, from which an aliquot was taken and dried to determine its concentration, that is grams per cubic centimeter. Thus in sampling, the tediousness of weighing was eliminated.

Analytical Work

1

Free Acids

Duplicate samples were taken, to which were added fifty milliliters of neutralized ninety-five per cent alcohol, three to four drops of phenolphthalein, and the whole brought to a boil. This alcoholic solution was then titrated with 0.1 N alcoholic potash solution until a pink color permanent for one minute was obtained.

Results	Lot 1	Lot 2
Free acids as oleic	54.0%	14.8%

Results are based on the alcohol extract being equal to 100 per cent.

2

Total Ether Solubles After Acid Digestion

Samples of the alcohol extract were taken and placed in conical flasks and fifty milliliters of standard sulfuric acid were added and the whole digested for one hour under a water reflux. The solution was transferred to a separatory funnel, cooled and ethyl ether added. The whole was shaken up, then allowed to separate into layers. The aqueous portion was drawn off into another funnel and extracted twice more with ether. The ether portions were combined and washed with water, then filtered and evaporated to dryness on the water bath, then weighed.

Results	Lot 1	Lot 2
	72.8%	75.3%

Results are based on the alcohol extract being equal to 100 per cent.

3

Total Fatty Acids

The total ether solubles residue from determination two was taken up in fifty milliliters of neutralized ninety-five per cent alcohol and phenolphthalein added. It was brought to a boil, then titrated with 0.1N alcoholic potash solution until the pink color obtained is permanent for one minute.

Results	Lot 1	Lot 2
Ether Soluble Fatty Acids as Oleic	32.4%	41.2%

Results are based on the alcohol extract being equal to 100 per cent.

4

Combined Fatty Acids

It is usual in practice for total Fatty Acids minus Free Fatty Acids to equal Combined Fatty Acids. A peculiar situation appeared at this point. In the case of solvent scoured wool, a greater amount of free acids was obtained than ether soluble fatty acids. This was quite contrary to what was expected. It was thought that the loss in fatty acids which occurred in the total fatty acids determination, might be due to the presence of volatile acids. The following determinations were made to clear up this point.

5

Total Volatile Acids

Samples of the original material being investigated, were placed in distillation flasks and an excess of sulfuric acid was added to each and the whole connected for distillation. The distillation was continued for one hour, the distillate being collected in standard alcoholic potash containing phenolphthalein. The excess alkali was then back titrated with standard sulfuric acid. The solution was evaporated to dryness and weighed. The potassium sulfate formed by the excess alkali and added acid was calculated, then subtracted from the weight of the residue, giving the weight of the potassium salt of the volatile acid. Knowing the exact amount of alkali needed to neutralize the volatile acids as well as the weight of the potassium salt formed, made it possible to calculate the approximate equivalent weight of the volatile acid, which was found to be 46 and which suggests formic acid.

Results	Lot 1	Lot 2
Volatile Acids equivalent weight 46.	1.5%	3.4%

Results are based on the alcohol extract being equal to 100 per cent.

The results were disappointing to say the least, the amount of volatile acid being insignificant compared to the amount of acid missing.

6

Total Water Soluble Acids

The aqueous portion remaining after determination two was retained and enough alcoholic potash was added to exactly neutralize the standard sulfuric used in determination two. This solution was then carefully evaporated to dryness on the water bath, then extracted thoroughly with several portions of boiling alcohol. Phenolphthalein was added to the alcoholic solution and the whole titrated with standard alcoholic potash. The neutralized solution was evaporated to dryness on the water bath and weighed. It was possible to calculate the equivalent weight of this water soluble acid from, the weight of the potassium salt formed and the known amount of potassium hydroxide necessary to form the salt. The equivalent weight was calculated to be 254 and the results are based on this figure.

Results	Lot 1	Lot 2
Water Soluble Acids equivalent weight 254	19.5%	13.3%

Results are based on the alcohol extract being equal to 100 per cent.

These water soluble acids, when expressed in terms of oleic acid and added to the total fatty acids for lot 1, equalled the free acids calculated to oleic.
19.5%=21.7% as oleic + 32.4%=54.1% of free acids Thus the discrepancy between the free acids determination and the ether soluble fatty acids determination is accounted for.

In the case of soap scoured wool, these water soluble acids appear to be in the free state only, since 13.3 per cent acid of equivalent weight 254 equals 14.8 per cent in terms of oleic acid. Therefore the ether soluble fatty acids obtained in determination three must occur only in the combined state, probably as soap.

7

Unsaponifiables

Samples of the original alcoholic extract were placed in conical flasks, alcoholic potash added and the whole digested under a reflux for several hours. The material was transferred to a casserole and neutralized with sodium bicarbonate, then evaporated nearly to dryness. At this point a small amount of previously extracted sand was stirred in, and the drying carried on to completion. The sandy mass was then transferred to an extraction thimble, which was in turn placed in a Soxhlet extractor. This material was extracted for six hours with ethyl ether, the ether portion then being filtered, evaporated to dryness and weighed.

Results	Lot 1	Lot 2
Unsaponifiables	20.0%	18.4%

Results are based on the alcohol extract being equal to 100 per cent.

8

Total Fatty Acids after Removal of the Unsaponifiables

The sandy residue from the determination of unsaponifiables was carefully leached with several portions of hot distilled water, sulfuric acid added in excess and the whole digested for an hour on the water bath. The solution was carefully transferred to a separatory funnel, cooled and extracted three times with ethyl ether. The combined ether portions were washed thoroughly with distilled water, then filtered, evaporated and dried on the water bath.

Results	Lot 1	Lot 2
Total Fatty Acids	53.0%	56.6%

Results are based on the alcohol extract being equal to 100 per cent.

The fatty residue from above was taken up in boiling neutral alcohol, phenolphthalein added and titrated with alcoholic potash.

Results	Lot 1	Lot 2
Total Fatty Acids as Oleic	52.5%	56.0%

Results are based on the alcohol extract being equal to 100 per cent.

The increase in total fatty acids after a thorough saponification and removal of the unsaponifiables, indicates the presence of a considerable amount of esters, probably esters of the higher alcohols or sterols. The reasonably close check for the two sets of results under this determination, indicates that those fatty acids combined with the unsaponifiable matter as esters are either oleic or a mixture of fatty acids, whose average equivalent weight is approximately the same as that of oleic acid.

9

Combined Fatty Acids as Esters

Fatty acids combined as esters, are found by difference. The difference between total fatty acids after removal of unsaponifiables and total fatty acids under determination two.

Results	Lot 1	Lot 2
	53.0%	56.6%
	32.4	41.2
Fatty Acids as Esters	20.6%	15.4%

Results are based on the alcohol extract being equal to 100 per cent.

10

Combined Fatty Acids as Soap (Sodium Oleate)

In the case of solvent scoured wool there appears to be no combined fatty acids in the form of soap, since all of the fatty acid obtained exists either in the free form or as an ester of the higher alcohols.

In the case of soap scoured wool there is an appreciable portion of the fatty acids existing in the form of a soap as sodium oleate. The amount of combined fatty acids is equal to the total fatty acid found in determination three.

11

Ash

Samples were carefully ashed in platinum and weighed.

Results	Lot 1	Lot 2
Ash	5.6%	13.3%

Results are based on the alcohol extract being equal to 100 per cent.

12

Ash Alkalinity

The platinum crucibles containing the ash were placed in beakers and carefully treated with warm distilled water, methyl orange added and the whole titrated, with standard acid.

Results	Lot 1	Lot 2
Milliequivalents acid used per gram of material	0.302	1.93

13

Metals, Fe, Al. Ca and Mg.

A large sample of the material being investigated was carefully ashed in platinum, then dissolved in warm distilled water. It was acidified with hydrochloric acid, the volume of the solution then being made up to the mark in a 250 ml. volumetric flask, with distilled water.

Iron and Aluminium

The acid aqueous solution (100 ml. of the above solution) was brought to a boil, a few drops of bromine water added and the boiling continued for a few minutes to drive off the excess bromine. Concentrated ammonia was added with stirring until the solution became definitely ammo-

niacal. It was boiled two to three minutes then allowed to stand a few minutes. This test proved to be negative meaning there was no iron or aluminium present.

Calcium Oxide

The solution remaining from the iron and aluminium determinations was heated to boiling and 10 ml. of ammonium chloride solution plus 10 ml. of saturated ammonium oxalate solution were added, followed by sufficient concentrated ammonia to make the solution definitely ammoniacal. After thorough stirring, the whole was then digested for one hour on the water bath. This test also proved to be negative.

Magnesium Oxide

The solution remaining from the calcium oxide determination was warmed and 10 ml. of sodium ammonium phosphate solution were added slowly with constant stirring. This was followed by 25 ml. of concentrated ammonia solution. The covered beaker was allowed to set four hours or more before filtration. This determination showed only a slight trace of magnesium oxide, which might be due to impurities in the chemical used.

14

Potassium Oxide

Another 100 ml. portion of the acid aqueous solution from above was evaporated to dryness, the residue then taken up with hot water and filtered. The solution was then brought to a boil, acidified with hydrochloric acid and a slight excess of barium chloride was added to remove sulfate ion which interferes with the determination of the potassium oxide. It was allowed to stand over night, then filtered, the precipitate washed and the filtrate plus washings were made alkaline with ammonia. Ammonium carbonate was added until no further precipitate was formed and also a few drops of ammonium oxalate. It was filtered and washed. The filtrate was carefully evaporated to dryness in a casserole, then heated just below redness till all of the ammonium chloride fumes were gone. It was cooled, and the residue dissolved in 25 ml. of hot water. One ml. of 60 per cent perchloric acid was added, then it was evaporated until nearly dry and when heavy fumes of perchloric acid appeared, it was cooled. Hot water, perchloric acid etc., treatments were repeated. When cool 35 ml. of alcohol were added and the whole filtered on a fritted glass crucible, washing with alcohol, drying and weighing.

Results	Lot 1	Lot 2
Potassium Oxide	3.5%	1.9%

Results are based on the alcohol extract being equal to 100 per cent.

15

Sodium Oxide

Another portion of the acid aqueous solution was placed in an Erlenmeyer flask and evaporated to dryness on the water bath. The residue was taken up in the smallest portion of water possible (15 ml.). Magnesium uranyl acetate reagent was added in excess, the number of ml. of reagent added being approximately ten times the number of mg. of sodium oxide present. The flask was partially immersed in water whose temperature was under 20° C. and the solution was vigorously stirred with a motor stirrer for 30-45 mins. The precipitate was then immediately filtered into a weighed fritted glass crucible using gentle suction. The flask and precipitates were washed with ninety-five per cent alcohol, the crucible then dried at 105-110° C. for thirty minutes.

Results	Lot 1	Lot 2
Sodium Oxide	0.0%	5.9%

Results are based on the alcohol extract being equal to 100 per cent.

16

Chloride Ion

A sample of the alcohol extract was digested for one hour with concentrated sulfuric acid, potassium sulfate and copper sulfate, as in a typical Kjeldahl digestion, in order to destroy the organic matter. The digest was taken up in a considerable amount of distilled water, the sulfate ion removed by the usual method, and the chloride ion precipitate with silver nitrate.

Chloride Ion	0.9%	0.6%

Results are based on the alcohol extract being equal to 100 per cent.

17

Sulfur as Sulfate

Another portion of the acid aqueous solution was made strongly acid with hydrochloric acid, heated to boiling and barium chloride added in slight excess. It was allowed to stand over night, then filtered, washed, dried, ignited and weighed as barium sulfate.

Results	Lot 1	Lot 2
Sulfur as Sulfate Ion	0.7%	0.4%

Results are based on the alcohol extract being equal to 100 per cent.

18

Nitrogen by Micro-Kjeldahl

Samples of the alcohol extract were analyzed for nitrogen by the Micro-Kjeldahl method through the cooperation of Milton Harris, A.A.T.C. & C. Research Associate at the Bureau of Standards.

Results	Lot 1	Lot 2
Nitrogen	2.1%	1.4%

Results are based on the alcohol extract being equal to 100 per cent.

Ash Alkalinity in Terms of K_2O and Na_2O

0.302 Milliequivalents of acid	Lot 1	1.5% K_2O
1.930 Milliequivalents of acid	Lot 2	0.9% K_2O and 5.9% Na_2O

Calculated Distribution of Salts in Ash

	Lot 1	Lot 2
Potassium Carbonate	2.2%	1.4%
Sodium Carbonate	...	10.1
Potassium Sulfate	1.3	0.7
Potassium Chloride	1.9	1.3
Total	5.4%	13.5%
Actual Ash	5.6%	13.3%

Tabulated Results

1.	Free Acids as Oleic	54.0%	14.8%
2.	Total Ether Solubles after Acid Digestion.	72.8	75.3
3.	Ether Soluble Fatty Acids as Oleic	32.4	41.2
4.	Combined Fatty Acids as Oleic		
5.	Total Volatile Acids (Equivalent Weight 46)	1.5	3.4
6.	Total Water Soluble Acids (Equivalent Weight 254)	19.5	13.3
7.	Unsaponifiables	20.0	18.4
8.	Total Fatty Acids after Removal of Unsaps.	53.0	56.6
		52.5	56.0
9.	Combined Fatty Acids as Esters	20.6	15.4
10.	Combined Fatty Acids as Soap (Sodium Oleate)	...	41.2
11.	Ash	5.6	13.3
12.	Ash Alkalinity K_2O	1.5	0.9
	Na_2O	...	5.9
13.	Fe, Al, Ca & Mg	None	
14.	Potassium as K_2O	3.5	1.9
15.	Sodium as Na_2O	...	5.9
16.	Chloride Ion	0.9	0.6
17.	Sulfate Ion	0.7	0.4
18.	Nitrogen	2.1	1.4

Lot 1 Solvent Processed.
Lot 2 Soap and Alkali Processed.

Composition of Alcohol Extracts From Analysis

	Lot 1	Lot 2
Free Fatty Acids as Oleic Acid (Water Insoluble)	32.4%	...%
Free Acids of Equivalent Weight 254 (Water Soluble)	19.5	13.3
Free Volatile Acids of Equivalent Weight 46 (Organic)	....	0.5
Soap as Sodium Oleate	....	44.4
Potassium Salt of the Volatile Acid	3.0	1.6
Sodium Salt of the Volatile Acid	....	3.1
Esters of Higher Alcohols	40.6	33.8
Potassium Chloride	1.9	1.3
Potassium Sulfate	1.3	0.7
Total	98.7%	98.7%

Lot 1 Solvent Processed.
Lot 2 Soap and Alkali Processed.

The composition of these alcohol extracts was calculated from the listed determinations, as follows:—

1. Free Fatty Acids as Oleic are equal to the ether soluble fatty acids (determination 3) for Lot 1, since there appeared to be no combined fatty acid as soap. In Lot 2 there was no free fatty acid, since all of the fatty acid was accounted for either as a soap or an ester of the higher alcohols.

2. Free Acids of Equivalent Weight 254 (water soluble) were found to be in the free state in both cases, having been determined under test one and also isolated under test six. For Lot 1, 19.5 per cent of this acid is equal to 21.7 per cent of acid calculated to Oleic. Therefore 32.4 per cent of fatty acids as oleic, plus this water soluble acid calculated to oleic, equals the free acids calculated to oleic, or the 54.1 per cent.

For Lot 2, 13.3 per cent of this water soluble acid equals 14.8 per cent of acid calculated to oleic, which is exactly what was obtained in the free acid determination (1).

Soap as sodium oleate is found by calculating all of the ether soluble fatty acids of Lot 2, as oleic (3), to the combined state or soap.

Potassium and sodium salts of the volatile acids are calculated from determinations 5 (volatile acids) and 12 (ash alkalinity).

Esters of the higher alcohols are obtained by adding unsaponifiables (7) to the combined fatty acids as esters (9).

Chlorides and sulfates of potassium are calculated from determinations 14, 16 and 17.

SUMMARY

One of the laboratory tests made to determine the efficiency of the mill cleaning process for raw or greasy wool, is an extraction test using ethyl alcohol. The amount of material extracted indicates the efficiency of the cleaning process. This investigation should assist in explaining the character of this extracted material, thus correcting any erroneous impression which may exist among textile processors, who may have believed that the extracted material consisted entirely of soap.

Approximately the same amount of alcohol extractable material can be obtained from solvent process scoured wool, which has had no contact with soap, as from wool scoured with soap and sodium carbonate. In the above cases, 1.1 per cent and 1.2 per cent respectively of alcohol extractable material was obtained, based on the dry weight of the wool. In the case of soap scoured wool, less than one-half of the alcohol extract is actually soap. The fact that there was no free fatty acid for the soap scoured wool, suggests that the free fatty acid in raw wool was neutralized by the added alkali in the scouring bowl to form soap.

Esters of the higher alcohols form a large part of the alcohol extract, the remainder being mostly free acids of equivalent weight 254. These free acids may possibly be connected with the nitrogen found in this alcohol extract. This suggests that the alcohol has removed actual wool substance.

A logical outcome of this investigation would be the development of a reliable method for determining soap in wool. Such a method would be of considerable value as a means of control for scouring processes.

ACKNOWLEDGMENT

This investigation was made possible by a grant from the Textile Foundation to the American Association of Textile Chemists and Colorists. We wish to express our appreciation for the aid given.

The Next Quarterly Research Number will appear April 4, 1938

A Note on

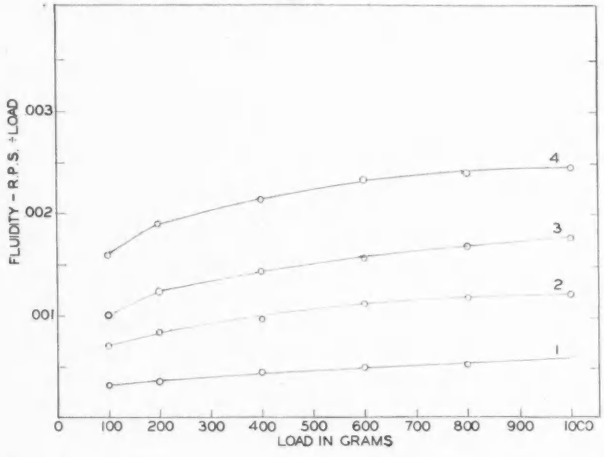
Vat Printing Pastes[1]

LELAND F. GLEYSTEEN[2]

THE present work constitutes a continuation of the researches on vat printing pastes undertaken by Dr. S. N. Glarum[3, 4]. Experiments have been begun to investigate the color yields obtained from printing colors containing various concentrations of thickening agents, particularly heat converted British gums of several different degrees of conversion. Gerber has published data[5], obtained by means of a step-photometer, showing influences of thickening agents on color values obtained in vat dye printing but it appears necessary to obtain further information before formulation of any general conclusions.

This paper is a report on the continuation of the work on Vat Printing Pastes previously carried on by Dr. Sivert N. Glarum and now being conducted by Dr. Leland F. Gleysteen. It is a research project of the A. A. T. C. C. which is being financed wholly by the Textile Foundation.

Fig. 1



The technique of preparing pastes, as in the case in which the gum-starch ratio was 1:1, has been found to be a most important factor in determining the properties of the finished paste.

For printing pastes in which the British-gum ratio is 2:1 or greater the behavior of pastes during cooking differs markedly from that of the pastes of higher starch content as described by Glarum[4]. All samples were prepared and cooked in an apparatus designed and described by Glarum[4]. Adequate stirring, thorough scraping of the walls of the vessel and relatively accurate means of determining the temperature *of the paste* are assured by this means.

LENGTH OF COOKING

Flow curves, obtained from measurements with a Stormer viscometer, as shown in Fig. 1, illustrate the effect of the length of cooking on a paste of the following composition:

Corn starch	4%
British gum (15% sol.)	16
Soda Ash	13½
Sulfoxylate	12
Glycerin	6
Water	48½
	100

All four pastes were heated to a temperature of 180° F. and the soda ash was added. Thereafter thickener number one was cooked at 200° F. for fifteen minutes, number two for thirty minutes, number three for forty-five minutes and number four for one hour. It will be observed that a quite short cooking gives the greatest consistency. It is obvious, therefore, that mills using a paste made up with a high British gum content (that is to say, two or more parts of British gum to one part of starch) will obtain maximum economy in the use of their thickeners by cooking for short periods of time.

TEMPERATURE OF COOKING

The effect of temperature of cooking on a paste of the above given composition is clearly shown by the flow curves of Fig. 2. Thickener number one was cooked at a

[1]This project is financed by an allocation from the Textile Foundation, Inc. This project is being carried out by the Research Committee of the A. A. T. C. C., the sub-committee on Textile Printing Pastes acting as advisors. This sub-committee is composed of William H. Cady, Chairman, Sivert N. Glarum, Arthur E. Hirst, Morell Mackenzie, Louis A. Olney, Robert E. Rose and Earle K. Strachan.

[2]Research Associate of the A. A. T. C. C. Through the courtesy of Brown University, the facilities of the Jesse Metcalf Chemical Laboratory have been made available for this work.

[3]S. N. Glarum, Am. Dyestuff Reptr. *25,* 150 (1936).

[4]S. N. Glarum, Am. Dyestuff Reptr. *65,* 437 (1937).

[5]H. Gerber, Melliand Textilberichte, *18,* 316 (1937).

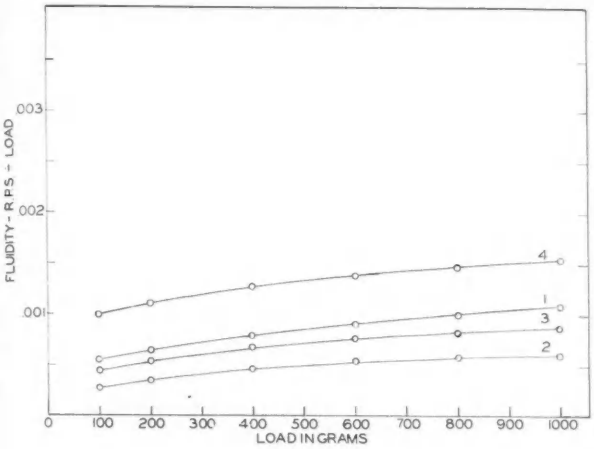


Fig. 2

temperature of 170° F., number two at 180°, number three at 190° and number four at 200°. In marked contrast to pastes of 1:1 British gum-starch ratio, it is seen that the optimum temperature is, in the present case, 175° F. In the average plant the starch kettles probably operate near this temperature. For pastes of higher starch content, however, best results, both in regard to maximum thickening and maximum color yield, are obtained at the highest temperature range which can conveniently be used, namely, 200°-210° F. This temperature range is not realized in most contemporary plant practice.

FURTHER CONSIDERATIONS

Some of the more important factors determining the cost of a printing color are:

The amount of dye required to produce a desired shade.
The amount of thickening agents required, starches, gums, dextrins, etc.
The amount of "Chemicals" (reducing agent, soda ash or potash) used.
The amount of hygroscopic agent and other assistants (oils, turpentine, etc.) employed.

This project has been limited, generally speaking, to the first two of these factors. The first factor is dependent upon most of the others, upon the method of preparing the paste, upon ageing conditions and perhaps still other variables. Providing other qualities of the print are not impaired, any feasible method of reducing, even in small degree, the amount of dyestuff required to do a given piece of work should represent a desirable saving. Those methods of cooking which produce maximum viscosity usually also produce good color yields[4]. Such evidence as is available seems to indicate that, given pastes of suitable flow characteristics, the less thickening agent the greater the color yield with the restriction that thickening agents of only one class be compared with each other[5]. Until much more is known regarding the mechanism of ageing it will be impossible to formulate, to say nothing of realizing, ideal ageing conditions.

Inasmuch as economy of time is often as important as economy of materials and sometimes even more important, and in view of the established correlation between flow characteristics and print quality, it would appear entirely logical to make routine viscometric readings on all pastes produced for vat prints to assure that they may be sent out possessing suitable flow properties.

The Use of Standard Dyeings in Tests for

Color-Fastness to Light[1, 2]

W. D. APPEL*

THE testing and rating of textiles with respect to color-fastness to light may be given a new significance as a result of the recent action of the Research Committee of the American Association of Textile Chemists and Colorists in adopting standard dyeings of known fastness and undertaking to make them available to all laboratories. These dyeings will enable the laboratories for the first time to express fastness on a common basis.

The standards are four in number and represent the minimum requirements for fastness classes 1, 3, 5, and 8 (in increasing order of fastness)[3]. The same standards have been adopted by the British and the German fastness committees and may be regarded as international. The selection of the intermediate standards, 2, 4, 6, and 7,

*Chief, Textile Section, National Bureau of Standards; Chairman, Sub-Committee on Lightfastness, A. A. T. C. C.

[1]Presented at the annual meeting of the American Association of Textile Chemists and Colorists in Philadelphia, Pa., December 4, 1937.

[2]Publication approved by the Director of the National Bureau of Standards of the U. S. Department of Commerce.

[3]1937 Year Book of the American Association of Textile Chemists and Colorists.

which are needed principally for classifying dyestuffs, is being held in abeyance pending further discussion by the committees abroad.

Since many potential users of the standards may not be familiar with the extensive work that has led to their selection[4], a brief statement of why such standards are necessary and how they can be used to the best advantage may be helpful.

The ideal test for color-fastness to light would require two things: first, apparatus with which the fading resulting from exposure in service can be duplicated in a short time; and, second, instrumental means for measuring the fading.

The first is approximated in available apparatus. For example, 20 or 40 hours of exposure in the commonly-used Fade-Ometer[5], a glass-enclosed carbon arc lamp apparatus, will produce the fading of textiles that may require as many days or weeks of exposure in service. However, the amount of fading resulting from exposure for a given number of hours even in the same apparatus varies from time to time because of variations in the temperature or humidity of the atmosphere about the samples, in the electric current supplied to the apparatus, or other factors. The time required to fade dyeings to the same extent in the apparatus in different laboratories varies by 20 or 30 per cent. In consequence, fading cannot be rated in hours of exposure required to produce a given amount of fading. The cost of the equipment needed to eliminate the variation in results appears prohibitive.

Fortunately, the relative fading of dyeings in the lamps is reproducible. Therefore, by exposing the materials to be tested and dyeings of known fastness at the same time, the fastness of the first may be rated by comparison with the fastness of the second. This procedure should lead to the same results in different laboratories using the standard samples of the Association even though the testing apparatus is not identical in fading action. One advantage of the standard dyeings is that their use does not involve a change in the familiar testing procedure.

The standards have been so chosen that they can be used in any of the common types of light tests, in accelerated laboratory tests, the standard sun test of the Association, or in the continuous daylight exposure test often used in England and Germany.

The second thing required by the ideal test is instrumental means for measuring the fading. Quantitative methods for this purpose are not available in most textile laboratories. Such methods are too slow for routine testing, though they can be used to advantage when visual judgment is uncertain. The changes in color of the standard dyeings of the Association provide a guide when the method of visual inspection is used. The fastness of most dyeings can be judged rapidly and satisfactorily with the aid of the standards.

For the present, then, the standard dyeings from a central source of supply seem to offer the simplest and most practical basis for standardizing the testing and rating of color fastness to light.

If the standards are to be used to the best advantage, the ways in which dyeings fade must be taken into consideration. Some dyeings start to fade in a short exposure but are still serviceable after long exposure. Others start to fade only after rather long exposure but fade rapidly thereafter. Dyeings may fade to about the same extent when exposed for one period of time but to different extents in others. The best practice in judging fastness seems to be to compare the samples and standards in at least two exposures, the first timed to show the initial fading and the second to show a more advanced stage of fading.

The standards of lower degrees of fastness, 1 and 3, show greater differences in rates of fading when exposed to sunlight (standard sun test) or to the diffused daylight in a room than to the higher intensities of laboratory lamps. If the intensity of the radiation on the samples in laboratory tests is decreased by placing them further from the source, the differences in fastness are more readily judged. The time required is longer, but it is still within the 24 hours required by much commercial testing.

Whether the standards from the central source need be exposed every time a test is made depends upon the reproducibility of the results in the apparatus to be used. Standards exposed one day probably can be used in judging tests made in the same apparatus under the same conditions for several weeks or even longer. Laboratories doing much testing probably will want to provide themselves with a considerable quantity of cloth which has the same fastness as the standards for use in every-day testing. In the settlement of disputes and in judging materials falling near the borderline of the fastness classes, the standards should be exposed simultaneously with the test samples.

Caution is necessary in the interpretation of the results of tests for fastness to light. The fact that dyeings have the same fastness classification does not mean that they will necessarily have the same fastness in service. The fastness in service depends upon so many factors that no single laboratory test for fastness to light can be correlated with days or weeks of use except in a very general way. However, tests for color fastness to light can be placed on a much firmer base than heretofore by the use of the standards, and more meaningful interpretations of the results can be given with their aid.

The value of the standards will depend upon the extent to which they are used and upon the intelligence with which they are used. Now that the standards are available, it rests with the laboratories to make them effective.

[4]The reports of the work of the American Association of Textile Chemists and Colorists are listed in the 1937 Year Book of the Association on pages 88, 89, and 90.

[5]Atlas Electric Devices Co., Chicago, Ill.

Report of the Rayon Crepe Research

IV—An Investigation of the Manufacture of Uniform Rayon Crepe Dress Fabrics

INTRODUCTION

THE purpose of this part of the Rayon Crepe Research was (1) to determine whether crepe fabrics made from regular deliveries of rayon yarn, processed on some of the best commercial equipment available and by methods which were representative of the best commercial practices, would contain any undesirable variations in pebble, and (2) to predetermine the quality and uniformity of finished fabric which could be obtained from the rayon yarns and methods of processing which were to be used subsequently in finding the effects of variables in each process in the manufacture of rayon crepe fabrics.

The Rayon Crepe Research was conducted jointly by the Throwsters Research Institute, Inc., and the A. A. T. C. C. The financing of the program was made possible through a grant from the Textile Foundation, Inc., to the A. A. T. C. C. and this grant was matched by contributions raised by the Throwsters Research Institute, Inc., from the industry and generous donations from their own treasury. W. F. Macia was the Research Fellow. Parts I, II, and III appeared in the November 1, 1937, Quarterly Research Number. Part IV as released by the committee is given herewith. Later parts will appear in future research numbers.

The work in each part of this program is so closely related that the readers are cautioned against drawing conclusions wholly on the basis of the results of the individual parts. The Rayon Crepe Research Committee will correlate all of the data and publish their conclusions in the last issue.

SUMMARY

Five hundred yards of rayon crepe fabric were made on some of the best commercial equipment and by representative commercial methods to determine (1) whether it was possible to make fabrics which were free from variations in pebble from regular deliveries of warp and filling yarns, and (2) to predetermine the quality and uniformity of finished fabric which could be obtained from the rayon yarns and methods of processing which were to be used subsequently in this research.

This investigation showed that it is possible to make rayon crepe dress fabrics free from objectionable variations in pebble, using yarn having the physical properties which tests showed were within the range found in ordinary commercial deliveries. (See Part No. 6 in this report for test results.)

It also showed that uniform fabrics could be made from the yarns provided for this research when processed by the methods adopted.

SCOPE OF THIS INVESTIGATION

The work in this investigation included the making of 500 yards of fabric, from normal shipments of warp and filling yarn in modern throwing, weaving, dyeing and finishing plants, under the close supervision of the research fellow.

The great amount of detail involved in this investigation precluded the possibility of experimenting with more than one type of warp yarn, one type of filling yarn and one fabric construction. The warp yarn used was from one lot and shipment; and the filling yarn was also from one lot and shipment. The warp and filling yarns were drawn at random from the regular production of the respective companies who donated the yarn for this research.

The fabric selected and made was a type particularly susceptible to variations in pebble elevation and filling bandiness. The construction was as follows:

	Warp Yarn	*Filling Yarn*
Type	Acetate Rayon	Viscose Rayon
Luster	Dull	Semi-Dull
Denier	150	200
Filaments	60	75
Twist	3.5	50
Ends per inch	Warp 96	Filling 48

Reed width, 47 inches

Weave—Plain, alternating 4 picks of "S" and 4 picks of "Z" Twist

CONTROL INSTRUMENTS

The instruments used in controlling and recording the conditions under which the yarns and fabrics in this investigation were processed were the same ones used in the surveys of the numerous throwing and weaving plants (Part No. 1 in this report). Periodic checks were made of the instruments to verify their accuracy.

GENERAL METHODS AND EQUIPMENT USED

All of the yarn that was thrown and the fabric that was woven, dyed and finished was processed in an average commercial manner in some of the best plants and on modern standard equipment.

The methods and formulae used as a basis for processing the yarns and fabrics in this research were not the standard practices of the plants in which the work was done but were selected from the surveys of the various plants (Part No. 1 of this report), by the Committee, or by the research fellow in cooperation with the staffs of the respective plants.

The plants in which the work was done were selected by the Committee because (1) they were representative of the industry, and (2) they were able to furnish the exacting control necessary for this type of research.

PREPARATION OF WARP YARN

Warping

The warp yarn was received at the weaving plant on two pound cones. A warp was made directly from the cones on a *Sipp-Eastwood horizontal automatic cone creel warper which was operated at 200 yards per minute. The warp consisted of 19 sections totaling 4740 ends and was 1300 yards long.

Slashing

The warp was sized on a *Johnson Rayon slasher. The details of this operation were as follows:

Number of drying cylinders—5.

Cylinder No. 1 was covered with cloth while the others were bare.

Circumference of drying cylinders—70 inches.

Temperature of drying cylinders:

1st—150° F.; 2nd, 3rd, 4th—170° F.; 5th—140° F.

Running speed of warp—18 to 20 yds. per min.

Stretch of yarn—3.0 per cent.

Warp Size Formula

The warp size formula used in this investigation was selected by the Rayon Crepe Research Committee after numerous inquiries into commercial formulae.

When this research began a study on this subject was being carried on by the U. S. Institute for Textile Research, but as this work was not completed, the Committee selected a formula containing the ingredients and approximate proportions generally used in commercial practice.

The choice by the Committee, however, was not intended to be a recommendation of the formula or ingredients.

The formula used in this investigation was as follows:

1600 lbs. of water
64 lbs. of gelatin
15 lbs. of softener (oil)
1 lb. of stearic acid

The characteristics of the gelatin were as follows:

Jelly-Strength—396 Viscosity—127

The viscosity and jelly strength was determined in accordance with the standard methods of the National Association of Glue Manufacturers, Inc. The jelly tests were made at a 7-1 concentration; i.e., on the glue basis.

The softener or oil was a mixture of cocoanut and sulfonated olive oils conforming to the following analysis:

Moisture	15%
SO_3	4%
Free Fatty Acid	7–8
Ash	4.5%
Unsaponifiable	Nil

It gave a stable light opalescent emulsion in water.

*The Committee has made no study of the comparative or respective merits of any particular make of machines used. They used the equipment which was available in the plants selected.

Preparation of Warp Size

The gelatin was steeped in cold water for four hours, then warmed gradually to 140° F. and stirred gently until it was completely dissolved. The stearic acid was melted, mixed with the oil then added to the gelatin. This solution was placed into the slasher and the same solution was used for replenishing.

Warp Characteristics

The sized warp had good cohesion, was of medium stiffness, flexible and wove excellently.

PREPARATION OF THE FILLING YARN

The filling yarn was received at the throwing plant in skein form packed in the usual ten pound bundles.

Soaking of Filling Yarn

After due consideration the Committee decided that the filling yarn in this research should be soaked by the standing bath or "tub method." Although this method has been displaced by machine soaking in many instances, it is still more extensively used in throwing plants than any other.

The filling soaking formula selected by the Committee contained ingredients and proportions corresponding closely to general commercial practice.

The filling soaking formula used in this investigation was as follows:

1¼ lbs. of gelatin.
1¼ lbs. of dextrine.
18 lbs. of oil.
Soft water to bring the total volume to 55 gallons.
110 lbs, of yarn.

The characteristics of the gelatin used in this formula were as follows:

Jelly-strength—372. Viscosity—132. pH—5.8.

The viscosity and jelly strength was determined in accordance with the standard methods of the National Association of Glue Manufacturers, Inc. The jelly tests were made at a 7-1 concentration; i.e., on the glue basis.

The pH determinations were made on the test samples after the jelly test had been made by remelting and determining the pH of the solution at 60° C. by a colormetric method (Lamott roulette comparator using brom thymol blue and chlor phenol red).

The dextrine used in this formula was manufactured from tapioca starch and conformed to the following specifications:

Color	Light canary
Solubility	99% plus
Specific Rotation (25° C.)	164
Dextrine by Polarization (25° C.)	87.5%
Dextrose	2.07%
Ash	0.10%

The oil used was a mixture of cocoanut and sulfonated olive oils conforming to the following analysis:

Moisture	15%
SO_3	4%
Free Fatty Acid	7–8
Ash	4.5%
Unsaponifiable	Nil

It gave a stable light opalescent emulsion in water.

Filling Soaking Procedure

Two filling soaking baths were used, one for the "S" twist yarn and one for the "Z" twist yarn. Exactly the same ingredients and proportions were used in each. The procedure used in preparing the baths was as follows:

The baths were started by running water into the tubs until about half the volume was reached. The gelatin and dextrine was then steeped in a separate container for a few minutes in cold water, gently stirred, then heated to 140° F. and stirred until fully dissolved. The solution was then poured through a filter into the baths. The oil was then poured through the filter into the baths and the tint which had been previously dissolved in water was filtered and added to the baths. Sufficient water was then added until the total volume was reached. The temperature was raised to 125° F. and the baths were thoroughly stirred with an electric propeller stirrer.

The skeins were placed in coarse mesh bags and submerged in the soaking baths for 30 minutes. They were then removed, allowed to drain three minutes and extracted until both "S" and "Z" yarns weighed approximately twice their original weights. The extracted skeins were gently shaken out manually and were hung on the poles of dryer trucks.

Drying Oven and Method Used

At the time that this work was being carried on there was some disagreement among technicians regarding a satisfactory temperature to use in drying rayon yarns and a research program is now being conducted by the U. S. Institute for Textile Research to determine facts on this subject.

The temperature used most extensively in throwing plants is approximately 180° F. whereas many technicians recommend 135° F. Because of the absence of facts on this subject, the Rayon Crepe Research Committee chose the arbitrary temperature of 150° F. as the base or control drying temperature for this research.

The drying oven used could be described as the truck type. Trucks having poles on which the skeins of yarn were suspended were pushed into the oven. The oven was maintained at 150° F. ± 5° F. The yarn was allowed to remain in the oven about three hours or until it contained 5.0 per cent moisture.

When the yarn was dry the trucks were removed and placed in the humidified winding room for 48 hours to allow the yarn to come to equilibrium with the atmosphere in which it was wound.

Winding

It was the purpose of this research to process yarn in an average commercial manner. The averages as found in the surveys of the numerous throwing and weaving plants were followed as closely as possible in processing the yarn in the mechanical operations, but in some instances it was necessary to depart slightly from the averages because the equipment in the plant in which the yarn was processed could not be altered to conform to all of the requirements.

The filling yarn in this part of the research was wound by the following method:

Make of winders	*Atwood
Make of swifts	*Atwood-wire adjustable hub
Weight on swift axles	12 ounces
Length of yarn under strain	19½ inches
Linear speed of winding (Yards per minute)	Minimum 100 Maximum 175
Atmospheric conditions (average)	Temperature 84.5° F. Relative Humidity 57.0%

Tension or Strain on the Yarn During Winding

Range Tension (Grams)	*Per Cent of Ends*
26 to 30	1.2
31 to 35	47.5
36 to 40	37.9
41 to 45	11.6
46 to 50	1.8

Twisting or Throwing

The filling yarn was twisted or thrown by the following method:

Make of twisters	*Atwood, double deck
Spindle speeds	10,000 R.P.M.
Length of yarn under strain	12 inches
Twist per inch	50
Flyer:	
Make	McHale
Type	4 arm, mill nut
Weight	No. 10 wire
Average atmospheric conditions	Temperature 86° F. Relative Humidity 55%

Tension or Strain on the Yarn During Twisting

Range Tension (Grams)	*Per Cent of Ends*
11 to 15	53.1
16 to 20	17.8
21 to 25	20.6
26 to 30	8.2
31 to 35	.3

Twist Setting or Steaming

The amount of steaming necessary to cause the yarn to lie dormant or refrain from untwisting or tangling depends upon a number of factors including: the denier of the

*The Committee has made no study of the comparative or respective merits of any particular make of machines used. They used the equipment which was available in the plants selected.

yarn, twist per inch, size and type of take-up shafts, the traverse of the yarn on the shafts and the type of steaming apparatus. For the purpose of this research, it was considered that a properly set yarn was one which reasonably resisted untwisting or kinking in all of the subsequent operations. The time, temperature and humidity used were chosen because they produced the best results with the apparatus and equipment used.

The twisted yarn was steamed in a *Lydon Electric steam box. The box was maintained at 180° F. dry bulb, and 175° F. wet bulb (89 per cent relative humidity), and the yarn was allowed to remain in the box continuously for three and one-half hours, under these conditions. The yarn was then removed and placed in the humidified redrawing room for twelve hours before redrawing.

Redrawing

The filling yarn was redrawn on to shipping spools by the following method:

Make of machines	*Atwood
Running speed of yarn (Yards per minute)	Minimum 140 Maximum 300
Length of yarn under strain	18 inches

Tension or Strain on the Yarn During Redrawing

Range Tension (Grams)	Per Cent of Ends
21 to 25	7.5
26 to 30	50.0
31 to 35	23.8
36 to 40	17.5
41 to 45	1.2

Average atmospheric conditions: Temperature 84° F. Relative Humidity 53.0%

The spools of yarn were wrapped, packed into cases and shipped to the weaving plant.

Copping or Quilling

The filling yarn was copped or quilled using the following specifications:

Make of machines	*Universal (Leesona)
Spindle speeds	2200 R.P.M.
Tension compensators	Flat wire with revolving Porcelain pulley
Average atmospheric conditions	Temperature 82.5° F. Relative Humidity 50.5%

Tension or Strain on the Yarn During Copping

Range Tension (Grams)	Per Cent of Ends
51 to 55	21.0
56 to 60	30.5
61 to 65	30.0
66 to 70	16.0
71 to 75	2.5

*The Committee has made no study of the comparative or respective merits of any particular make of machines used. They used the equipment which was available in the plants selected.

Weaving

Five hundred yards of fabric were woven by the following method:

Make of loom	*Crompton & Knowles
Type	2 x 1 automatic shuttle change
Running speed	140 picks per minute
Number of harnesses used	6
Type of let-off motion	rope
Ends per dent in reed	3
Ends per heddle-eye	1
Draw	1-6-4-2-5-3
Spread of yarn in reed	47 inches
Average warp tension as recorded by *Crompton & Knowles warp tension meter	50 ends read 50 to 60
Average atmospheric conditions	Temperature 85.0° F. Relative Humidity 57.0%

DYEING AND FINISHING

A large portion of the Rayon Crepe Research was devoted to an investigation of the effects of variations in the individual operations in dyeing and finishing. Previous to this investigation, however, hundreds of yards of experimental fabric were dyed in an ordinary commercial manner.

In a research program of this type it was not possible to determine the average methods used in dyeing and finishing rayon crepe dress fabrics without making a survey of representative plants in the dyeing industry. Since this could not be done within the scope of the Rayon Crepe Research, it became necessary to rely upon the knowledge and experiences of some of the members of the Committee and the wide experiences of the dyeing and finishing plant in which the work was done.

In this particular investigation the experimental pieces were dyed and finished by the methods used commercially by the company which furnished the dyeing and finishing facilities to this research. The methods used conformed closely to general trade practices.

The entire processing was followed closely by the research fellow.

The detailed methods used in dyeing and finishing the pieces were as follows:

Marking and Beaming

The pieces were carefully marked for identification with indelible pigment, were sewed together and beamed into continuous lengths of 250 yards each.

Boil-Off

The scouring was done on a continuous boil-off machine consisting of two separate units. The first unit consisted of a rectangular tank approximately 30 feet long, 6 feet wide and 6 feet deep in which the bath was maintained at a temperature of 180° F. The purpose of the first unit was to shrink the fabric in flat form. It was equipped so that the fabric was passed through with a minimum

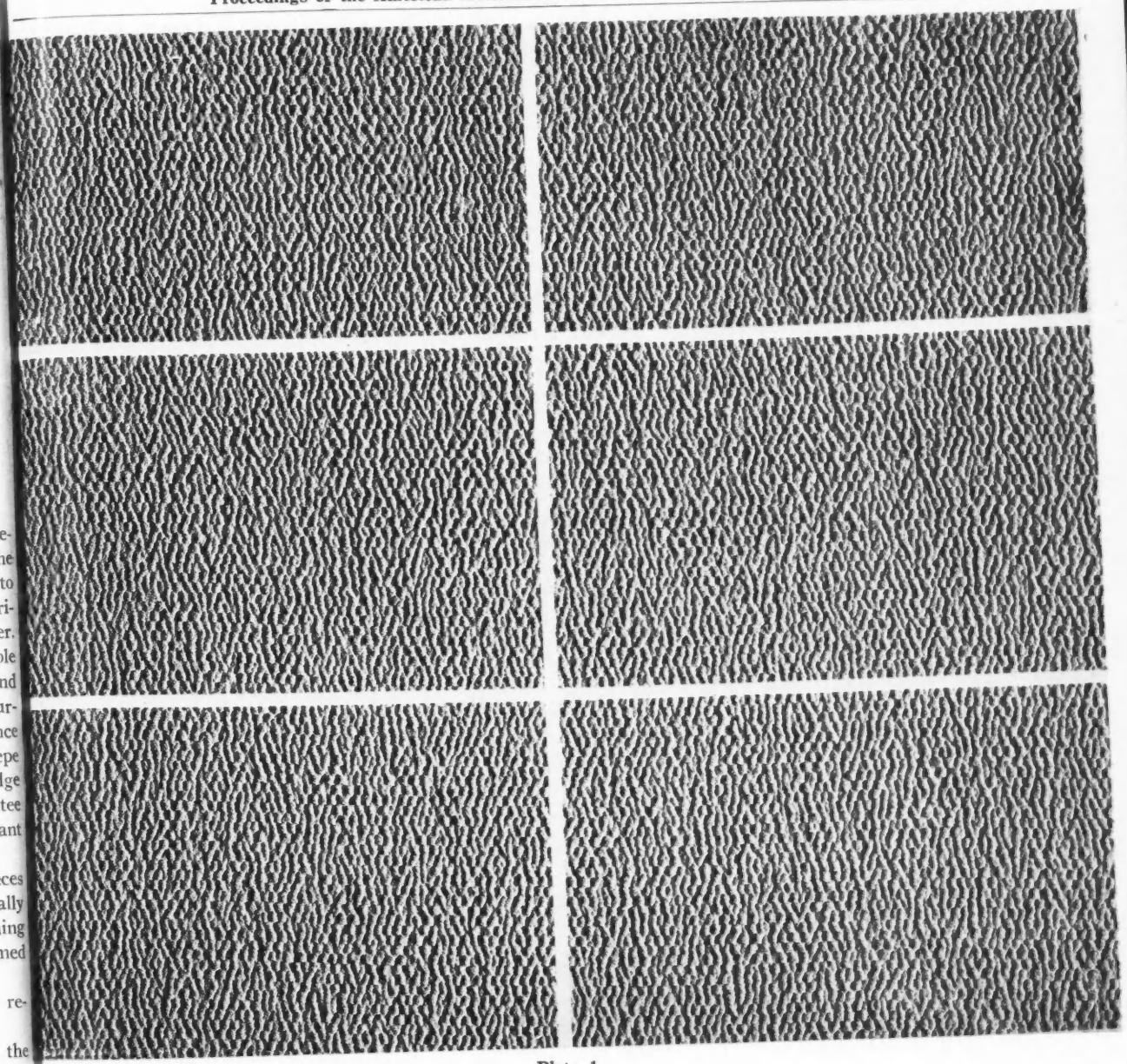


Plate 1

of tension either in the warp or filling-wise direction. The cloth was fed into the bath from above at one end and was drawn off at the other. The only tension used was that necessary to draw the fabric through the bath and lift it out of the tank. At no time was the fabric allowed to sink to the bottom of the tank or accumulate at any one point. The fabric was drawn off the first unit still in flat form, then converted through eyes into rope form and fed into the second unit. The second unit consisted of several individual compartments through which the fabric was continuously passed in rope form. The first individual compartments contained a ½% soap solution, whereas the last units were used entirely as rinses. Upon completing the cycle the fabric was fed from the machine on to a table. The goods were in the boil-off machine for a total of 25 minutes.

The pieces were individually segregated and examined to determine whether any further processing might be necessary to assure complete removal of tint and soilings. These pieces required no further processing.

Dyeing

The pieces were dyed in an ordinary beck in rope form. They were entered into a cold bath consisting only of water, circulated a few minutes to insure complete wetting

out then the following dyes and ingredients were introduced:

For Dyeing the Acetate Rayon

1/2% Setacyl Direct Blue G (41%)
2/100% Calconese Yellow GC
3/100% Calconese Red Violet R Double
2/100% Artisil Red SY

For Dyeing the Viscose Rayon

1/10% Direct Pure Blue RC
4/100% Brill Benzo Violet B

For exhausting 1/10 per cent of Glauber's salt and as an assistant 25/100 per cent of Naccanol were used in the bath. All of the percentages were calculated on the weight of the fabric.

The temperature was gradually raised up to the boil and the pieces were circulated for fours hours to insure complete penetration of the dye. After this period the dye bath was flooded off and the goods rinsed first in warm water then cold water for one-half hour. After the application of a finishing solution in the last rinse, the pieces were removed from the box and sent for extraction.

Extracting

The goods were extracted until the bulk of the water was removed.

Drying

The pieces were dried on a standard dye-house loop or slack type dryer, the goods being continuously carried through the dryers in festoon form.

Re-Beaming

The pieces were sewn together again, spread out and again wound into rolls of 250 yards in length in preparation for the tentering process.

Tentering

The goods were tentered on a tentering frame which was equipped with wet steam jets which released wet steam upon the fabric at the entering end of the frame. The goods were passed over hot steam coils which dried them while being spread to 39 inches in width. After tentering they were finally segregated into the individual pieces, rerolled and examined.

FINISHED FABRIC EXAMINATION

The finished pieces were carefully examined by the Committee. The fabric had a medium elevation of pebble which was generally uniform in character. No objectionable variations in pebble were visible within the individual pieces and no differences were discernible from piece to piece. The consensus of opinion was that the pieces could be considered commercially perfect from the standpoint of pebble uniformity.

This investigation demonstrated that it was possible to make commercially perfect cloth insofar as pebble uniformity is concerned from the yarns used if reasonable control is exercised over each operation from the first in the throwing plant to the last in the dyeing and finishing plant.

This yarn and the methods used therefore represent suitable standards by which to judge the variations of yarn and methods which are the subject of the subsequent parts still to be reported upon.

The accompanying photographs in Plate No. 1 show the type of fabric made and the character and elevation of pebble.

Evaluation of Crease-Resistant Finishes for Fabrics*

HERBERT F. SCHIEFER

ABSTRACT

CERTAIN flexural characteristics of representative woven fabrics before and after the application of commercial crease-resistant finishes were measured using three different methods, namely the creasing-angle, flexometer, and modified compressometer methods. Specimens of paper, Cellophane, cotton, rayon, worsted, and rubber, which are recognized to vary in resistance to creasing over a great range, were also measured by the three methods and the results used for comparison.

The crease-resistant finishing treatments increased the energy required to deform the cloth specimens, that is, increased the stiffness of the cloths. This increase exceeded 100 per cent for some of the cloths. The energy of recovery was also increased by these treatments, the increase ranging from 20 to 100 per cent. Neither of these two quantities can be taken individually as a measure of crease resistance. However, the ratio of the latter to the former, here termed "resilience", is related to the resistance to creasing. The resilience of nearly all of the cloths, as determined by each of the three test methods, was increased by the crease-resistant finishing treatments.

The three test methods described can be used to evaluate the improvement given by crease-resistant finishes, using the measurements on the cloths before treatment as the basis of comparison. The methods should be valuable in

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systematic studies of the effect of different finishing treatments and in determining the effects of relative humidity, temperature, and other factors on the different finishes. Method 1, however, does not give a critical measure of stiffness, a property that is greatly affected by crease-resistant finishes and should be measured. The resilience as determined by these methods is dependent upon the testing conditions, as for example the magnitude of the maximum load in method 3. It may be found necessary in test methods of this type to vary the maximum load with the thickness or weight of the fabric to be tested.

I. INTRODUCTION

Most textile fibers are deficient in natural resilience. Articles made from them tend to become wrinkled and to lose their shape and desired appearance. It has been found possible in recent years to improve the resilience of textiles by chemical processing, and crease-resistant fabrics are now available. The "anticrease" process of Tootal, Broadhurst, Lee, Ltd., which is being applied in the United States as well as in England, was the first to attract attention. In it the fabric is impregnated with a synthetic resin. This development in the industry has led to a need for a quantitative method for evaluating the improvement of the product and of the resilience of fabrics in general. The work reported in this paper was undertaken to determine the flexural characteristics of woven fabrics before and after the application of commercial crease-resistant finishes. The results are compared with the flexural characteristics of a series of samples which have not had crease-resistant treatments, but which are recognized to vary in resistance to creasing over a considerable range.

The flexural characteristics of the samples were measured by three different methods. The results are presented and discussed in this paper.

II. MATERIALS AND TEST METHODS

1. Samples

Representative woven fabrics ranging from light-weight voile to heavy suiting cloth, before and after the application of crease-resistant finishing treatments, were furnished for the work by several manufacturers. These fabrics were made from cotton, rayon, and linen. Specimens of paper, Cellophane, cotton, rayon, worsted, and rubber, having crease resistances varying over a great range, were added to the list of samples for comparison. Specimens of the materials were conditioned by exposure for several days in an atmosphere of 65 per cent relative humidity and a temperature of 70° F, and they were tested under these conditions.

2. Creasing-angle Method

The specimens tested were 2 inches long and ½ inch wide with the long dimension in the warp or in the filling for tests in these respective directions. Each specimen was folded by bringing the two ends into coincidence, thereby forming a loop at the middle. The two ends were held together with a pair of tweezers and the loop of the specimen was inserted between two parallel plates and placed under a load of 1.00 pound. The load was removed at the end of 3 minutes, and the specimen was picked up with the tweezers and suspended freely at the middle over a horizontal wire of small diameter, approximately 1 mm. At the end of 3 minutes the horizontal distance between the two ends was measured and the angle at the vertex, where the specimen was folded, was calculated. An angular scale, graduated in degrees, placed directly back of the test specimen, is a convenience in reading the angle directly. A device of this kind, developed by Barnard, is shown in Figure 1.

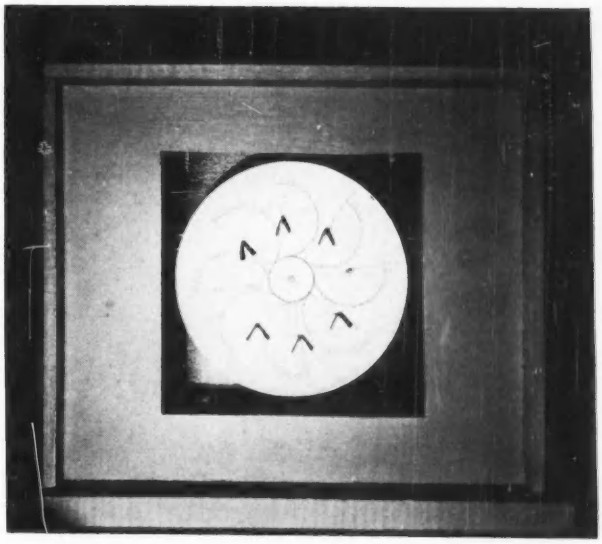

Fig. 1—Multiple Protractor for Measuring the Creasing Angle

Eight specimens can be suspended on it at a time. The disk on which the specimens are suspended can be rotated by means of a crank to bring any one of the eight specimens into position for measurement, that is, to align one edge of the specimen with the zero angle line and the other edge to indicate the magnitude of the creasing-angle.

In this test method the ends of the specimens droop an amount which depends not only upon the magnitude of the load and the time it is applied to the fold, but also upon the weight, thickness, and type of construction of the cloth. The angular deflections of the ends of the various specimens tested vary over a great range because of these inherent differences between the cloths. For example, the ends of a specimen of a thin sheet of rubber which shows no crease after the application of the load, droop more, even before the load is applied to the specimen, than the ends of a specimen of an easily creased cotton fabric after the application of the load to the fold. To take account of this natural drooping of the ends of each specimen, the angular deflection was determined both before and after the application of the load to the fold. The ratio

of the angle at the vertex of a specimen after the load is applied to the fold to the angle before the application of the load is here designated the "resilience ratio."

Various modifications[1] of this method for measuring the effect of crease-resistant treatments have been reported in the literature. In one arrangement the specimen is folded as already described and then compressed by a dead weight for several minutes, after which the specimen is placed on edge, with the fold in a vertical position, on the surface of mercury. The angle of the fold is measured after a specified period. The result by this method is influenced by the condition of the mercury surface. Also, it is not possible to determine the effect of the treatment on the stiffness of the cloth. Since the stiffness is greatly affected by the treatments and should be evaluated, the procedure of floating the creased specimen on mercury was not used in this investigation.

3. Flexometer Method

Two specimens, each 4 inches long and 1¾ inches wide with the long dimension in the warp or in the filling for tests in these respective directions, were mounted in opposite angles formed by the two intersecting vertical plates of the flexometer[2] shown in Figure 2. One of these plates

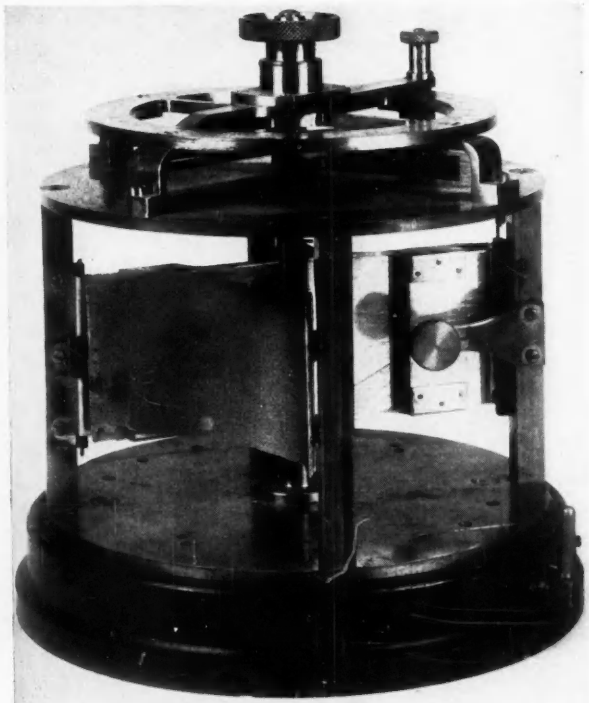

Fig. 2—Flexometer

is cut along the intersecting axis and each half is fastened to the frame of the instrument. The other plate is attached to a spindle and can be rotated freely about the axis of intersection of the two plates. The top of the spindle carries six calibrated cantilever springs mounted radially 60 degrees apart. A hub having six spokes which can be rotated by means of a knob is located above the six springs. By placing a pin in a spoke and rotating the hub the spring located directly below the spoke is deflected. The deflection of the spring exerts a torque on the spindle and causes the plate to rotate. The magnitude of this torque is equal to the torque exerted by the pair of test specimens as they resist being folded between the two plates. The magnitude of the applied torque is ascertained from the deflection of the calibrated cantilever spring. Readings were taken at definite angles between the plates, first for decreasing and then for increasing angles, according to the procedure described in Bureau Research Paper RP555. Successive readings were taken as rapidly as possible, the time required for one reading being about 15 seconds. The energy expended in folding the specimens through a given angle is equal to the product of the average torque and the change in the angle between the two plates. The total energy expended in folding the specimens to a minimum angle between the plates was determined, as well as the energy recovered when the specimens were allowed to unfold. The minimum angle to be employed was calculated from the thickness of the material using the equation $\beta = 430$ h, where β is the angle in degrees and h is the thickness in inches measured with a gage having a circular foot 1 inch in diameter and exerting a pressure of 1 lb. sq. in. The ratio of the energy recovered to the energy expended is here termed the "flexural resilience."

The work recovered after keeping the specimens folded at the minimum angle for 5 minutes, and the work done and recovered in refolding and unfolding the specimens after they were allowed to remain unfolded for 5 minutes were also measured. These data did not contribute to the evaluation of crease resistance and accordingly are not reported here. Apparently, 5 minutes was too short a period of time to allow for readjustments in the specimens.

4. Compressometer Method

A very sensitive modified compressometer, illustrated in Figure 3, was designed and used for measuring the flexural properties of light-weight woven fabrics. The instrument has a circular presser foot, 1 inch in diameter, which is fastened to the top of a helical spring through a ball-and-socket connection. The foot may be lowered or raised by means of a rack and pinion, thereby increasing or decreasing the load which the presser foot exerts upon a specimen placed on an anvil beneath it. This load is indicated by the dial indicator which registers the elonga-

[1] C. Amick, Crease-Resisting Fabrics, American Dyestuff Reporter, Vol. 24, pp. 554-7; 662-4; 645-8; 665 (1935).
W. Matthaes, Lilienfeld Rayon: Crease Resistance, Kunstseide, Vol. 18, pp. 292-293 (1936).
Society of Dyers and Colourists, Vol. 52, No. 1, p. 38; Vol. 52, No. 8, p. 117 (1936).
Crease-Resistant Textiles Without Resins, Silk & Rayon, Vol. 11, p. 46, January 1937.
K. Quehl, Staple Fiber Fabrics: Crease Resistance, Melliand Textilberichte, Vol. 18, pp. 241-2 (1937).

[2] Herbert F. Schiefer, The flexometer, an instrument for evaluating the flexural properties of cloth and similar materials, BS J. Research *10*, 647-57 (1933) RP555.

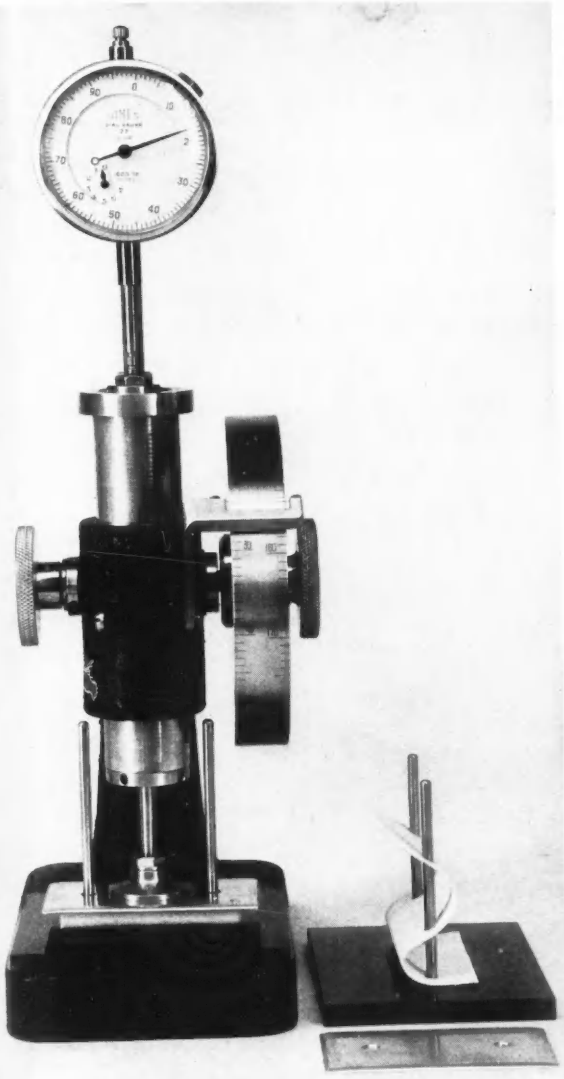

Fig. 3—Modified Compressometer

tion of the calibrated helical spring. The vertical displacement of the rack is indicated on the scale engraved upon the circumference of a disk which is fastened to the axis of the pinion. The vertical displacement of the foot is equal to the displacement of the rack minus the elongation of the spring. The rack and pinion must be uniform, because any irregularities in them produce non-uniform vertical displacements and necessitate either engraving a non-uniform scale on the disk or applying a correction.

The specimens tested were 3 inches long and 2 inches wide, with the long dimension in the warp or in the filling for tests in these respective directions. Two rows of three holes each were punched in each specimen. The holes were spaced 1 inch apart in the long direction and 1½ inches in the short direction. The specimen was mounted through these holes in an accordion fashion consisting of two loops on a special support, as illustrated in Figure 3. An aluminum plate weighing 0.02 pound was placed on this specimen. The support and specimen were placed underneath the presser foot of the compressometer. The height, in inches, of the accordion loops was determined first for increasing loads up to 0.60 pound and then for decreasing loads, readings being taken at 0.05, .10, .15, .20, .30, .40, and .60 pound. Successive readings were taken as rapidly as possible, the time required for one reading being about 10 seconds. The energy, in inch-pounds, expended during loading and that recovered during unloading were calculated, using the expression 0.025 (3a+2b+2c+3d+4e +6f—20g), where a, b, c, d, e, f, and g are the heights of the accordion loops at the above loads, respectively. The ratio of the energy recovered to the energy expended is here termed the "compressometer flexural resilience."

The data obtained after allowing the specimen to remain compressed for 5 minutes at the maximum load of 0.60 pound and after allowing it to remain uncompressed for 5 minutes did not contribute to the evaluation of crease resistance, agreeing with the flexometer method in this respect. However, the time of application of the load of deformation may be as important as the magnitude of the load. For example, a sample of worsted cloth in which the loops were compressed continuously for 3 months by a load of only 0.02 pound showed more pronounced creases of a more permanent nature, judged by visual appearance, than the creases produced by a load of 0.60 pound acting for only 5 minutes.

A method in which the test specimen is folded in a series of accordion loops and compressed by a dead weight has been used by Sommer[3]. The height of the loops under a small initial load, the height of the loops when compressed by a dead weight, and the final height of the loops after the weight was removed were used in evaluating a fabric. The value obtained decreased nonlinearly with the magnitude of the dead weight used. Specific details regarding this method and the time of application of the dead weight are not given.

III. RESULTS AND DISCUSSION

The results of the tests are given in Table 1.

Specimens 1 to 11, inclusive, are a representative group of cotton, rayon, and linen cloths ranging from light to heavy weight. Measurements were made on these cloths before and after crease-resistant finishing treatments. The results on these cloths indicate the effect of the crease-resistant treatment. Specimens 12 to 17 included paper, Cellophane, cotton, rayon, worsted, and rubber, which have not had crease-resistant treatments but which have crease resistances varying over a great range, as can be judged from the appearance of these specimens in Fig. 4. They also vary in stiffness over a great range. The measure-

[3] *Textilprufung auf neuen Wegen.* Abstract of a paper delivered at a meeting of the German Engineering Society, by Prof. H. Sommer, Director, Fibrous Division, National Institute for Testing Materials, Berlin, on September 25, 1936.

TABLE 1

Effect of crease-resistant finishing treatments on cloths measured by three methods

[Specimens 12 to 17, inclusive, are a series having crease resistances varying over a great range. W and F indicate the warp and filling directions, respectively. The units for work in methods 2 and 3 are different, to conform with those previously used for the flexometer and compressometer.]

Specimen	Thickness	Designation	Treatment	Direction	Method 1			Method 2			Method 3		
					Angle		"Resilience ratio"	Work done	Work recovered	"Flexural resilience"	Work done	Work recovered	"Compressometer flexural resilience"
					Before applying the load (1)	After applying the load (2)	(3)	(1)	(2)	(3)	(1)	(2)	(3)
	inch				deg.	deg.	per cent	ergs	ergs	per cent	in. lb.	in. lb.	per cent
1	.007	Cotton voile	not treated	W	148	39	26	43×10	18×10^2	41	34×10^{-4}	9×10^{-4}	26
			not treated	F	128	57	35	39	19	48	33	10	30
			treated	W	152	71	47	59	28	48	51	16	31
			treated	F	148	85	57	62	36	57	53	18	34
2	.008	Cotton voile	not treated	W	144	63	44	44	22	50	63	26	42
			not treated	F	137	50	36	48	21	44	70	28	40
			treated	W	134	81	60	64	35	55	100	32	32
			treated	F	129	70	60	58	35	60	91	34	37
3	.009	Cotton warp and rayon filling dress fabric	not treated	W	137	46	34	74	39	53	111	36	32
			not treated	F	144	55	38	80	47	59	119	32	27
			treated	W	137	70	51	109	65	60	131	48	37
			treated	F	128	74	58	109	76	70	138	45	33
4	.012	Spun rayon dress fabric	not treated	W	121	60	50	82	52	64	129	38	30
			not treated	F	103	46	45	63	38	60	114	34	30
			treated	W	180	123	68	187	103	55	197	70	35
			treated	F	110	65	59	76	53	70	119	52	44
5	.015	Spun rayon dress fabric	not treated	W	134	57	43	94	55	58	189	41	22
			not treated	F	108	63	58	67	41	61	144	44	31
			treated	W	152	87	57	240	122	51	245	84	35
			treated	F	128	81	63	109	72	66	159	73	46
6	.026	Cotton plaid	not treated	W	157	59	38	106	47	44	178	41	23
			not treated	F	152	55	36	125	52	41	144	43	30
			treated	W	180	80	44	193	88	45	218	60	28
			treated	F	180	79	44	178	79	44	176	55	31
7	.005	Rayon dress fabric	not treated	W	180	61	34	181	68	38	142	29	20
			not treated	F	140	45	32	85	35	41	96	28	29
			treated	W	164	106	65	171	104	61	197	67	34
			treated	F	130	75	58	111	75	67	130	39	30
8	.016	Rayon suiting	not treated	W	137	72	53	125	74	60	190	57	30
			not treated	F	140	72	51	179	100	56	210	70	33
			treated	W	148	79	53	154	93	60	202	72	36
			treated	F	148	84	57	241	138	57	234	85	40
			not treated	W	180	87	48	203	113	55	279	49	18
			not treated	F	128	93	73	69	46	66	134	56	42

9	.013	Rayon jacquard	not treated	W	180	87	48	203	113	55	279	49	18
			not treated	F	128	93	73	69	46	66	134	56	42
			treated	W	180	131	73	454	203	66	386	179	46
			treated	F	180	128	71	174	69	55	215	69	32
10	.020	Spun rayon suiting	not treated	W	162	54	34	168	80	48	304	50	16
			not treated	F	144	49	33	225	107	48	321	79	25
			treated	W	144	80	56	315	156	49	278	109	39
			treated	F	162	92	57	408	203	50	398	136	34
11	.019	Linen suiting	not treated	W	180	55	30	511	164	32	399	79	20
			not treated	F	180	47	33	414	122	29	325	64	20
			treated	W	157	60	55	413	251	61	371	141	38
			treated	F	152	59	47	231	144	62	268	105	39
12	.003	Bond typewriting paper	not treated	a	180	19	11	1360	273	20	567	137	24
			not treated	b	180	26	15	800	179	22	513	113	22
13	.002	Cellophane	not treated	a	180	26	14	274	84	31	265	36	14
			not treated	b	180	29	16	211	95	45	262	25	11
14	.005	Cotton lining	not treated	W	180	65	36	302	108	34	255	33	13
			not treated	F	180	57	32	259	91	25	214	29	14
15	.004	Rayon lining	not treated	W	180	78	43	96	47	49	189	28	15
			not treated	F	180	81	45	74	42	57	111	26	23
16	.018	Worsted necktie lining	not treated	W	180	123	68	286	221	77	323	208	64
			not treated	F	180	140	78	287	244	85	322	230	71
17	.012	Sheet rubber dental dam	not treated	—	61	55	90	78	61	79	138	125	91

Fig. 4—Specimens having Crease Resistances varying over a great range.

Top row: Appearance of specimens before wrinkling.
Middle row: Appearance of specimens after being compressed and crushed into a small ball.
Bottom row: Final appearance of specimens after being straightened out.

ments on them serve as a criterion of the relative crease resistance of the cloths before and after treatment. The data in Table 1 serve as a basis in comparing the three test methods used.

The values reported for each test method in the columns numbered (1) are related to the stiffness of the cloths; those in columns numbered (2) are related to the energy of recovery; and those in columns numbered (3) are related to the resilience.

The data in Table 1 are plotted in Fig. 5 to show the over-all effect of the crease-resistant treatments and for a comparison of the three test methods. The results for method 1 are plotted in the first column; those for method 2 are plotted in the second column; and those for method 3 are plotted in the last column. The values reported in the columns numbered (1) in Table 1 are plotted in the top row of Fig. 5; those in columns numbered (2) are plotted in the second row; and those in columns numbered (3) are plotted in the bottom row. The values for the treated specimens are plotted as abscissa and those for the specimens before treatment are plotted as ordinate. The 45-degree diagonal is drawn in each plot to indicate whether or not the treatment increased or decreased the quantity plotted, a point falling below or above the diagonal indicating an increase or a decrease, respectively. A point which falls on the diagonal indicates that the treatment had no effect on the quantity plotted. The data for specimens 12 to 17, which have not had crease-resistant treatments, are plotted on the 45-degree diagonal for ease of comparison with the results for the specimens before and after the crease-resistant treatment.

With only a few exceptions, the treatments increased the energy required to deform the cloth specimens, that is,

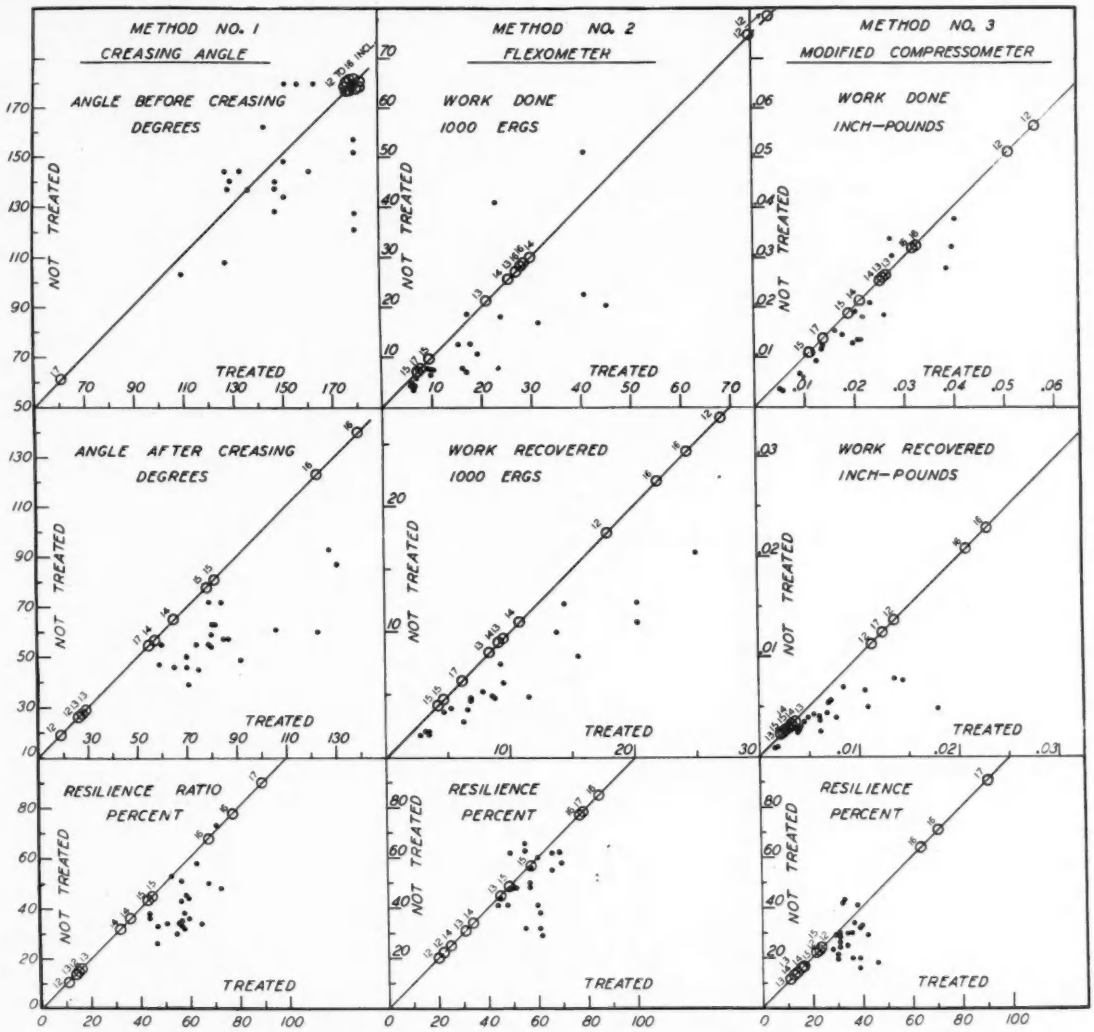

Fig. 5—Effect of Crease-Resistant Treatments on cloths as measured by three test methods. (Solid points indicate specimens 1 to 11 before and after treatment. Open circles, specimens 12 to 17, are plotted on the 45-degree diagonal for ease of comparison and indicate the fabrics having crease resistances varying over a great range without a treatment.)

increased the stiffness of the cloths. This increase exceeded 100 per cent for some of the cloths. Specimen 11, a linen fabric, is an exception.

Since the stiffness of the cloth is greatly affected by the crease-resistant treatments, the evaluation of this characteristic may be very important, at least for some of the uses for which these cloths may be intended. Method 1 does not give a critical measure of stiffness. Many of the specimens which are known to vary greatly in stiffness, in particular specimens 12 to 16, inclusive, are not differentiated by this method. This is a disadvantage of the method.

The energy of recovery of each fabric, including the linen specimen 11, was increased by the crease-resistant treatments. The increase ranged between 20 and 100 per cent. The percentage increase in the energy of recovery of the treated to the untreated specimens of the same cloth is no doubt a measure of the improvement resulting from the treatment. The magnitude of the angle after creasing, method 1, which has been used as a criterion of the resistance to creasing, is not necessarily a measure of crease resistance. According to such a criterion, specimen 17, which is a thin sheet of rubber and has practically no tendency to assume a permanent crease under a load, would be considered very poor compared to many of the other specimens which actually show a marked crease after the application of a load. For the same reason, the magnitude of the energy recovered in methods 2 or 3 cannot be taken as a criterion of the resistance to creasing of woven fabrics.

The ratio of the energy of recovery to that expended, designated "resilience," is related to the resistance to creasing. For example, specimens 12, 13, and 14, having low crease resistances, were found to have low values of resilience, whereas specimens 16 and 17, having high crease resistances, were found to possess high values of resilience. Using the resilience of these specimens as a criterion, the specimens 1 to 11, as a group, are superior to specimens 12, 13, and 14, but inferior to 16 and 17. Although the resilience of nearly all of the fabrics, as determined by each of the three test methods, was increased by the crease-resistant finishing treatments, as a group these fabrics are inferior to fabrics made from fibers having high natural resilience. These conclusions were also borne out by visual observation of the appearance of these specimens after the various tests. The general improvement in resilience imparted by the crease-resistant finishing treatments is well established, however, and still greater improvement may be expected to result from more extensive researches.

A closer examination of the results for resilience given in Table 1 indicates that the finishing treatments did not increase the resilience of all fabrics. The value for specimen 9 tested in the filling direction is a notable exception. The stiffness of this specimen was increased by a very large percentage in the filling direction. The warp and filling of this specimen were structurally very different. The increase in resilience for the warp and the decrease for the filling are no doubt related to the construction of this specimen and this behavior may also be expected in other specimens. In fact, the stiffness in the warp direction of specimens 4 and 5 was increased by a large percentage by the treatment and the resilience determined with the flexometer, method 2 in the table, was decreased. The increase in resilience indicated by methods 1 and 3 for these specimens justified a closer examination of the methods, specimens, and results obtained.

Check tests by the flexometer method indicated that the decrease in resilience is real. Check tests by the modified compressometer method, although somewhat variable, still indicated a higher resilience for the treated specimens. Additional tests were made using different maximum loads. These tests indicated a dependence of the resilience upon the maximum load, somewhat analogous to the results found by Sommer, to which reference was made earlier in this paper. Similar tests were made for specimen 2 by the modified compressometer method. The results, given in Table 2, show that the resilience increased with the maximum load used. Furthermore, the resilience of the treated fabric was slightly greater than that of the untreated fabric at a low maximum load, whereas at a high maximum load the reverse was indicated.

These results show that the reversal in resilience between methods 2 and 3 may be attributed to the difference in their testing conditions. In methods 1 and 3 the specimens are compressed by a constant maximum load, regardless of the weight or thickness of the specimens. In method 2, however, the specimens are folded to a minimum angle of such magnitude that equal stresses are produced in the extreme fibers of homogeneous isotropic specimens. Although fabrics cannot be considered as homogeneous isotropic specimens, the testing procedure in method 2 of folding the specimens to a minimum angle, which is determined from the thickness of the specimen, is more rational than applying a constant maximum load to all the specimens as in methods 1 and 3. For in deforming a specimen by a series of increasing loads, as in method 3, the total work expended on the specimen is equal to the summation of the products of the applied load and the distance through which the load acts. Thus, in testing two specimens which differ greatly in stiffness by applying the same maximum load, the deformation depends upon the stiffness, and the ratio of the energies expended or recovered is not necessarily the same as the ratio of the energies expended or recovered when these two specimens are deformed to the same degree, as in method 2, by applying a variable maximum torque proportional to the stiffness. Unfortunately, specimens 2, 4, and 5 were considerably wrinkled during shipment. Great care was exercised in sampling, but variations, which could not be detected by visual inspection, may have existed between the samples selected for test. Therefore, the variations in resilience by methods 2 and 3 for the specimens 2, 4, and 5 cannot be attributed definitely to the differences in the condition of test between methods 2 and 3.

The three test methods described can be used to evaluate the improvement given by crease-resistant finishes, using the measurements on the cloths before treatment as the basis of comparison. They should be valuable in systematic studies of the effect of different finishing treatments and in determining the effects of relative humidity, temperature, and other factors on the different finishes. Method 1, however, does not give a critical measure of stiffness, a property that is greatly affected by crease-resistant finishes

TABLE 2

Effect of maximum load used in the modified compressometer method on the flexural characteristics of cotton voile before and after treatment with crease-resistant finish.

Maximum load	*Specimen*	*Work done*	*Work recovered*	*Resilience*
lb.		in. lb.	in. lb.	per cent
0.30	Not treated	32×10^{-4}	5×10^{-4}	16
	Treated	46	8	17
.40	Not treated	41	8	20
	Treated	66	15	23
.50	Not treated	47	14	30
	Treated	80	18	23
.60	Not treated	63	26	42
	Treated	100	32	32

and should be measured. The resistance to creasing of fabrics in general is related to the resilience. The resilience as determined by these methods is dependent upon the testing conditions, as for example, the magnitude of the maximum load in method 3. It may be found necessary in test methods of this type to vary the maximum load with the thickness or weight of the fabric to be tested.

The user of fabrics, however, who probably judges crease resistance primarily by the changes in the appearance of the fabrics, is not concerned so much with the effects of finishing treatments on a particular fabric as with the resistance to creasing of different fabrics regardless of the treatment they may have received in the course of manufacture. Although the test methods described in this paper indicate whether or not an improvement in crease resistance results from a finishing treatment, they may not clearly indicate the relative merits of the fabrics of different compositions and constructions from the viewpoint of the user. For this there appears to be needed first a standard pro cedure for subjecting fabrics to creasing forces in a manne and under conditions that can be correlated with norma use of the fabrics, and second, a method for evaluatin the changes in appearance the fabrics undergo in this trea ment. Work on the development of an optical method o test for this purpose is being undertaken in the Textil Section of the National Bureau of Standards.

The author acknowledges his indebtedness to Mr Kenneth Barnard of the Pacific Mills and chairman of th "Committee on Test Methods for Crease Resistance an Permanent Finishes" of the American Association of Tex tile Chemists and Colorists, and to Dr. D. H. Power who is also a member of the committee, for suggestion and interest in this work. Grateful acknowledgment is als made to Röhm & Haas Company, Pacific Mills, and Toota Broadhurst & Lee, Limited, for furnishing material fo testing.

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